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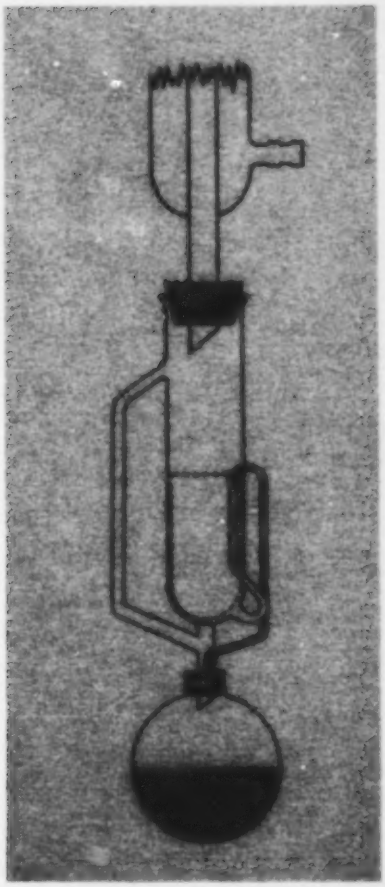

Compendium of Waste Leaching Tests

Report EPS 3/HA/7
May 1990

Canada

Environment Canada
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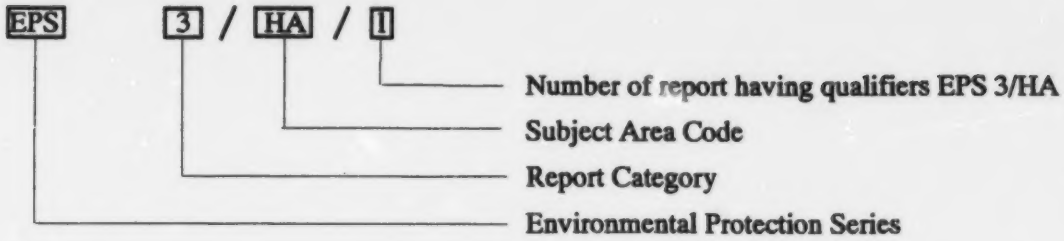

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by the

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ABSTRACT

This compendium is a layperson's introduction to leaching tests. It describes leaching in terms of concepts rather than mathematical equations, introduces a classification and uniform nomenclature for leaching tests, and includes a glossary and a comprehensive bibliography. As such, it gives the novice an overview of leaching test purposes and experimental features and the interpretation of test results while providing the expert with a unique list of references.

RÉSUMÉ

Ce compendium est destiné à la personne non initiée, afin de l'introduire aux essais de lixiviation. Ce document présente la lixiviation en termes de concepts plutôt que sous forme d'équations mathématiques, et il offre une classification et une nomenclature uniforme des essais de lixiviation, un glossaire et une bibliographie exhaustive. En tant que tel, il donne au profane un aperçu des objectifs des essais de lixiviation, des particularités expérimentales et de l'interprétation des résultats d'essai, tout en constituant pour l'expert une liste unique de références.

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GLOSSARY

Accuracy:	The degree to which a measurement equals the true value being measured.
Advective transport:	The movement of water under the influence of an hydraulic gradient.
Aliquot:	A known fractional part of a defined quantity.
Blank:	Specimen or leachate made of similar material and composition, but with the species of interest absent.
Channelling:	The tendency for water to find open, continuous pathways that offer less resistance to flow.
Chelating agent:	A ligand that attaches at two or more sites on a central metal ion.
Chemical equilibrium:	A reversible state in which there is no net particle or energy flow between phases or species.
Chemical equilibrium model:	A generalized mathematical description of a chemical system assuming equilibrium conditions.
Chemical stability:	The ability to resist change in chemical composition.
Contaminant:	Typically, an undesirable (e.g., hazardous or radioactive) constituent that renders another substance impure.
Desorption:	Release of adsorbed substances from a solid surface to a liquid phase.
Dissolution:	Transfer of a species from the solid to the liquid phase through chemical or physical interaction of the soluble species with the solvent.
Durability:	The ability of solidified waste forms to resist physical wear and chemical attack.
Effective porosity:	The fraction of the total void space through which water can percolate.
Effluent:	A liquid stream flowing in what is defined to be the outwards direction.
Electroneutrality:	The state in which a solution of dissolved ions is electrically neutral.
Elution:	Removal of adsorbed substances by the washing action of a leachant.
Fraction leached:	The mass of constituent in the leachate divided by the total mass of constituent originally present in the sample.
Hydraulic conductivity:	A proportionality constant with the dimensions length divided by time, which relates the water velocity or flux to the hydraulic gradient in a porous medium.

Hydraulic gradient:	The difference in hydraulic potential between two points, divided by the distance of separation.
Mechanistic models:	Detailed descriptions of leaching processes based on mathematical equations that represent fundamental mechanisms.
Molecular diffusion:	The movement of molecules under the influence of a difference in chemical potential.
Monolithic:	Massively solid, single, uniform.
Near subsurface:	Uppermost portion of the Earth's crust to the depth of excavated trenches used for the burial of wastes.
Partitioning:	The division of a chemical constituent among different phases.
Photolytic:	Change brought about by the action of radiant energy, including visible light.
Porosity:	The property of a material related to the amount of void space present, or the ratio of the void volume to the total volume.
Precision:	The degree to which the same measurements performed repeatedly are in agreement.
Proctor:	A standard laboratory compaction method for soil or soil-like material.
Radioactivity:	The property of certain elements that causes them to spontaneously emit radiation (consisting of alpha particles, electrons, neutrons, gamma rays, or X-rays) by the disintegration of the atomic nuclei.
Radionuclide:	An atom that exhibits radioactivity.
Redox potential (Eh):	The potential developed as an element is transformed from one oxidation state to another.
Release:	Mass of constituent in the leachate divided by the total mass of the sample.
Solid waste:	The fraction of a multiphase waste without any free liquid phase.
Spike:	A known amount of a constituent deliberately added to an aliquot of sample or leachant.
Steady state:	Constant net flux of a constituent between phases or components of a system.
Surface area of contact:	The amount of surface area contacted by a leachant.
Tortuosity:	The ratio of the length of a sinuous pathway between two points to the length of a straight line between the points.
Transport:	The conveyance of chemical constituents in the liquid and gas phases of a solid matrix.

Waste form:	The stable, solid body formed by the solidification, melting, or consolidation of waste materials, meeting specifications for emplacement.

SUMMARY

This compendium is a layperson's introduction to leaching tests. It describes leaching in terms of concepts rather than mathematical equations, introduces a classification and uniform nomenclature for leaching tests, and includes a glossary and a comprehensive bibliography. As such, it gives the novice an overview of leaching test purposes and experimental features and the interpretation of test results while providing the expert with a unique list of references.

In general, a leaching test involves contacting a waste material with a liquid to determine which components in the waste will dissolve in the liquid. The compendium presents a conceptual model of leaching mechanisms, including water flow, diffusion, and chemical reactions. These concepts are useful to illustrate how different mechanisms may limit leaching in the field and in laboratory leaching tests.

Leaching tests used in the hazardous and radioactive waste fields are surveyed and separated into two broad categories on the basis of whether or not the leachant is renewed: extraction tests (leachant not renewed) and dynamic tests (leachant renewed).

Extraction tests include all tests in which a specific amount of leachant is contacted with a specific amount of waste for a certain length of time, without leachant renewal. At the end of the contact period, the leachate is separated from the waste and analyzed. Extraction tests are further divided into four subcategories:

1) agitated extraction tests
2) nonagitated extraction tests
3) sequential chemical extraction tests
4) concentration buildup tests

Dynamic tests include all tests in which the leachant is continuously or intermittently renewed to maintain a driving force for leaching. Dynamic tests provide information about the kinetics of contaminant mobilization. Information is generated as a function of time, and attempts are often made to preserve the structural integrity of the waste form. These two factors lend this category to the investigation of more complex mechanisms of leaching.

Dynamic tests can be further divided into subcategories according to how the interface between the waste and the leachant is defined. Tests in which individual waste particles are used to define the interface are called serial batch tests. Tests in which a

characteristic dimension of the waste, such as the external geometric surface area or the geometric surface area perpendicular to flow, is used to define the interface include

1) flow-around tests
2) flow-through tests
3) soxhlet tests

Protocols selected for presentation satisfy one or more of the following criteria: 1) the protocol is sponsored by a recognized organization, 2) it is a voluntary or regulatory standard, 3) it is a publicly available protocol, or 4) representative results are published in the literature.

A brief history of each protocol, its intended purpose, and any noteworthy features of the method are reviewed. Specific details of the test variables are presented in tabular form, and supporting references for each type of test are listed in the bibliography. The following protocols are reviewed:

- Extraction Procedure Toxicity Test
- Leachate Extraction Procedure
- Toxicity Characteristics Leaching Procedure
- Quebec R.s.Q.
- California Waste Extraction Test
- French Leach Test
- American Society for Testing and Materials D3987
- Equilibrium Extraction
- Multiple Batch Leaching Procedure
- Materials Characterization Center - 1,2,3,4,5
- Sequential Chemical Extraction
- Standard Leaching Test, Procedure C, University of Wisconsin
- Multiple Extraction Procedure, Method 1320
- Monofill Waste Extraction Procedure
- Graded Serial Batch
- Sequential Batch Extraction
- Waste Research Unit Repetitive Shaker Test
- Standard Leaching Test, Cascade Test
- International Atomic Energy Agency Dynamic Leach Test
- International Standards Organization Leach Test
- American National Standards Institute/American Nuclear Society 16.1

- Dynamic Leach Test
- American Society for Testing and Materials Column Method
- Standard Leaching Test, Column Test

Seven experimental variables common to all extraction and dynamic tests are discussed in detail to assist the reader in selecting and interpreting existing protocols. The seven variables are 1) sample preparation, 2) leachant composition, 3) method of contact, 4) liquid-to-solid ratio, 5) contact time, 6) temperature, and 7) leachate separation.

Finally, interpretation considerations are presented, organized in four sections dealing with data examination, data reduction, time-independent analysis, and time-dependent analysis.

1 INTRODUCTION

In general, a leaching test involves contacting a waste material with a liquid to determine which components in the waste will dissolve in the liquid. The liquid, prior to contact with the waste, is called the leachant; after contact, it is called the leachate (Figure 1). Endless variations can be introduced by changing test variables, such as leachant composition, method of contact, liquid-to-solid ratio, contact time, and temperature, to investigate the chemical and mass-transport phenomena controlling the extent and rate of leaching.

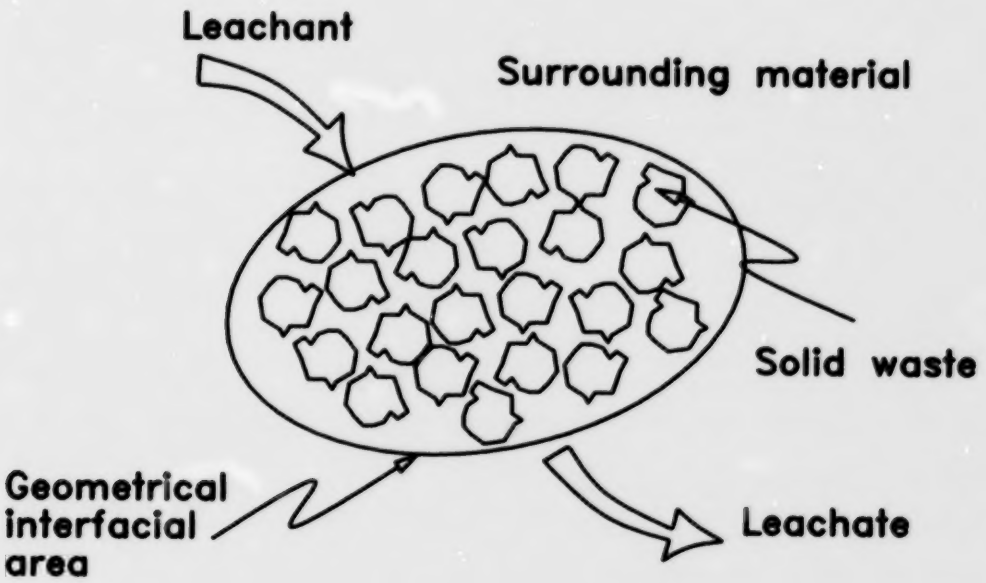


FIGURE 1 WASTE LEACHING SYSTEM

Leaching tests have a wide range of objectives, the most common of which are presented in Table 1. For several of these objectives, including

- identification of leachable constituents,
- classification of hazardous wastes,
- evaluation of process modifications,

TABLE 1 OBJECTIVES OF LEACHING TESTS

Objective	Description
Identification of leachable constituents	Determine which constituents of a waste are subject to dissolution upon contact with a liquid
Classification of hazardous wastes	Compare wastes against performance criteria for classification of wastes as hazardous or nonhazardous
Evaluation of process modifications	Determine if modifications to a waste-generating process result in a less leachable waste
Comparison of waste treatment methods	Determine whether a given waste treatment method/process results in superior containment of contaminants
Quality control in waste treatment	Verify the efficiency of a treatment process using a simple pass/fail criterion
Design of leachate treatment systems	Obtain a typical leachate to perform treatability experiments
Field concentration estimates	Express leaching over time (e.g., to be used as a source term in groundwater modelling)
Parameter quantification for modelling	Quantify partition coefficients and kinetic parameters to be used in transport modelling
Risk assessment	Estimate potential impact of waste disposal on the environment.

- comparison of waste treatment methods,
- quality control in waste treatment, and
- design of leachate treatment systems,

leaching tests are used to provide information about the constituent concentration or the constituent release from a solid waste under reference test conditions.

For the three remaining objectives:

- field concentration estimates,
- parameter quantification for modelling, and
- risk assessment,

a leaching test is used to provide information about constituent release under conditions that more closely approximate the actual disposal site. This information may subsequently be used in mathematical models to predict long-term leaching.

This document is organized as follows. A generic description of the leaching process is presented in Chapter 2. Many different leaching test methods were identified and reviewed by surveying the hazardous and radioactive waste literature, and these tests are classified and broadly defined in Chapter 3. The main test variables are discussed in Chapter 4, and selected protocols are described in Chapter 5. In Chapter 6, the temporal relationship between short-term laboratory tests and long-term field leaching is discussed. Finally, Chapter 7 considers how the results of various leaching tests can be expressed and how this expression influences the selection of a test for a given application.

This compendium was written to provide, in layperson's terms, a concise introduction to leaching tests. The waste leaching test literature comprises several hundred documents of all types, often not easily accessible. Publications originate from two scientific communities, dealing with hazardous and radioactive wastes, that have had little interaction and, consequently, have developed different nomenclatures and symbols. Reading research reports and scientific papers is often counterproductive because they are written for specialists and deal with specific problems.

This document is short, describes leaching in terms of concepts rather than mathematical equations, introduces a classification and uniform nomenclature for leaching tests, and includes a glossary and a comprehensive bibliography (the bibliography is organized according to the main test categories defined in Chapter 3). As such, it gives the novice an overview of leaching test purposes and experimental features and the interpretation of test results while providing the expert with a unique list of references.

2 DESCRIPTION OF THE LEACHING PROCESS

Leaching tests were initially developed to assess the short-term environmental impact of solid waste disposal in landfills. After disposal, the solid waste may come in contact with a leachant, such as rainwater, open surface water, or groundwater. Although attempts are made to isolate waste materials to prevent them from coming into contact with water, this is rarely possible. The result is that some of the constituents in the waste may dissolve into the leachant. Since the hydrologic cycle (Figure 2) involves a continual cycling of water between the atmosphere, surface water, and groundwater, the leaching of solid wastes and subsequent transport of dissolved waste constituents has far-reaching implications for the quality of the environment.

The kinds of solid wastes that are of greatest concern with respect to leaching in the near subsurface include incinerator, fly, and other combustion ashes; sludges and cakes from physical and chemical wastewater treatment operations; contaminated soils; foundry sands; mine tailings; tank bottom sludge; dredged sediments; and low and medium level radioactive wastes. These wastes are disposed of in the form of dry powders, slurries, and sludges, or as waste forms, and may contain a wide range of organic and inorganic constituents. The constituents that are potentially hazardous to the environment are termed contaminants and their presence in potable water should not exceed drinking water quality standards (e.g., Canadian drinking water guidelines are given in Table 2).

Leaching occurs when the contaminants in a waste come into contact with a leachant. The manner in which this contact occurs is determined by the physical and engineering properties of the disposed waste, such as water content, porosity, homogeneity, and hydraulic conductivity, and the corresponding properties of the material surrounding the waste.

Contact can occur by leachant flowing around the waste (Figure 3a), leachant flowing through the waste (Figure 3b), or a complex combination of these methods. Water will mainly flow around a porous waste if the hydraulic conductivity of the waste is much smaller than that of the surrounding material. Water may be forced to flow through the waste if the situation is reversed. Of equal importance is the hydraulic gradient across a waste material, which together with the hydraulic conductivity determines to what extent the contaminants will be exposed to leachant.

Once contaminants have been contacted by a leachant, leaching may ensue. Leaching encompasses the physical and chemical reactions that mobilize a contaminant, as well as the mechanisms of transport that carry the contaminant away from the waste.

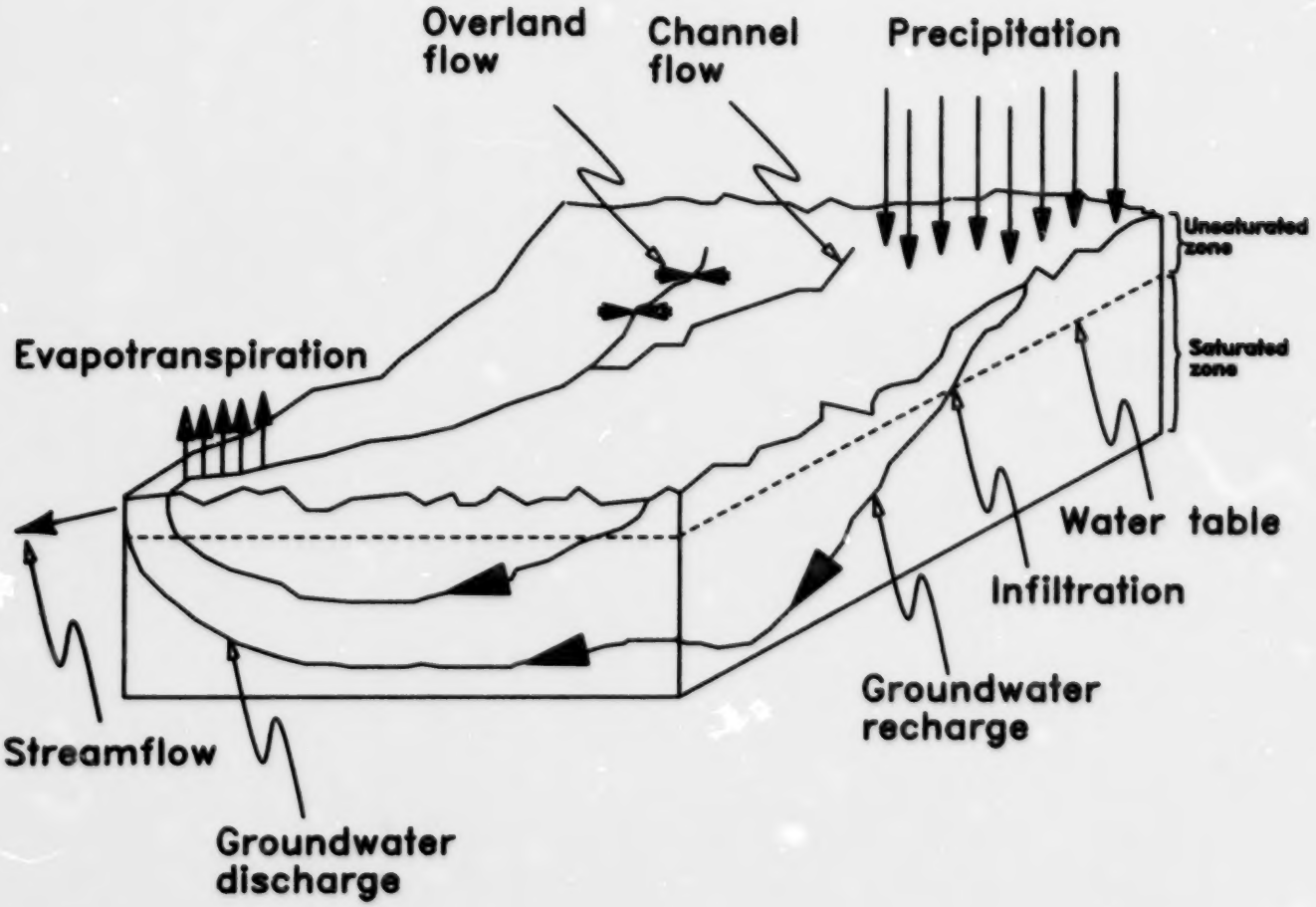
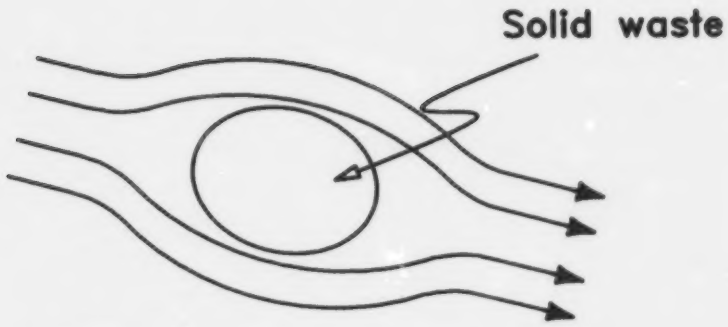


FIGURE 2 SCHEMATIC OF THE HYDROLOGIC CYCLE

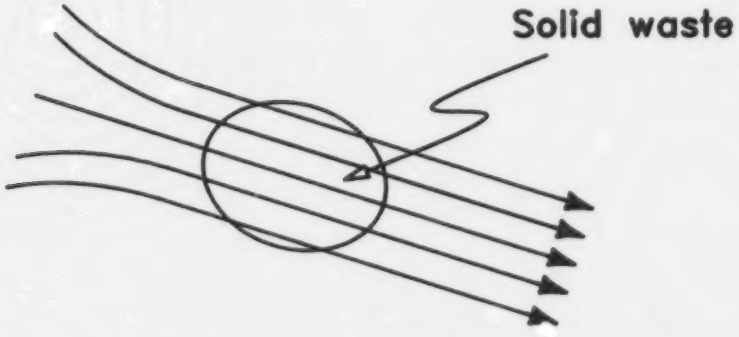
TABLE 2 GUIDELINES FOR CANADIAN DRINKING WATER QUALITY(1)

Parameter	Maximum Concentration	Parameter	Maximum Concentration
Alachlor	-	Heptachlor + Heptachlor Epoxide	0.003
Aldicarb	0.009	Iron	<0.3
Aldrin + Dieldrin	0.0007	Lead	0.05
Antimony	-	Lindane	0.004
Arsenic	0.05	Linuron	-
Asbestos	-	Malathion	0.19
Atrazine	0.06	Manganese	<0.05
Azinphos-methyl	0.02	MCPA	-
Barium	1.0	Mercury	0.001
Bendiocarb	0.04	Methoxychlor	0.9
Benzene	0.005	Methyl-parathion	0.007
Benzo(a)pyrene	0.00001	Metolachlor	0.05
Boron	5.0	Metribuzin	0.08
Bromoxynil	0.005	Nitrate	10.0
Cadmium	0.005	Nitrilotriacetic Acid (NTA)	0.05
Carbaryl	0.09	Nitrite	1.0
Carbofuran	0.09	Odour	-
Carbon tetrachloride	0.005	Paraquat	0.01
Chlordane	0.007	Parathion	0.05
Chloride	<250	PCBs	-
Chlorobenzene; 1,2-di-	0.003	Pesticides (total)	0.1
Chlorobenzene; 1,4-di-	0.001	pH	6.5-8.5
Chlorophenol; 2,4-di-	0.0003	Phenols	-
Chlorophenol; penta-	0.03	Phorate	0.002
Chlorophenol; 2,3,4,6-tetra-	0.001	Picloram	-
Chlorophenol; 2,4,6-tri-	0.002	Selenium	0.01
Chloropyrifos	0.09	Silver	-
Chromium	0.05	Simazine	0.01
Colour	<15 TCU	Sodium	-
Copper	<1.0	Sulphate	<150
Cyanazine	0.01	Sulphide (as H(2)S)	<0.05
Cyanide	0.2	2,4,5-T	0.02
2,4-D	0.1	2,4,5-TP	-
DDT + metabolites	0.03	Taste	-
Diazinon	0.02	TCA(i)	-
Dicamba	0.12	Temephos	0.28
1,2-Dichloroethane	-	Temperature	<15°C
1,1-Dichloroethylene	-	Terbufos	0.001
Dichloromethane	0.05	Tetrachloroethylene	-
Diclofop-methyl	0.009	Toluene	<0.024
Dieldrin + Aldrin	0.0007	Total dissolved solids	<500
Dimethoate	0.02	Toxaphene	-
Dinoseb	-	Triallate	0.23
Dioxins	-	1,1-Trichloroethane	-
Diquat	0.07	Trichloroethylene	-
Diuron	0.15	Trifluralin	-
Endrin	-	Trihalomethanes	0.35
Ethylbenzene	<0.0024	Turbidity	1 NTU
Fluoride	1.5	Uranium	0.1
Furans	-	Xylenes	<0.3
Gasoline	-	Zinc	<5.0
Glyphosate	0.28		
Hardness	100		

Note: values are in mg/L unless specified otherwise
TCU = true colour units (platinum cobalt scale)
NTU = nephelometric turbidity unit



a) Water flowing around the waste



b) Water flowing through the waste

FIGURE 3 EXTREME WATER FLOW REGIMES

To proceed with a discussion of these phenomena, it is necessary to introduce the two scales that can be used to describe leaching. At the macroscopic scale, leach rates apply to the geometrical interface that separates the solid waste from the surrounding material (Figure 1). At the microscopic or pore scale, leaching is described at the interface that separates individual particles (Figure 4).

In nonporous waste forms such as glasses and ceramics, where there are no voids within the waste, leaching is the result of interfacial exchanges at the outer surface by dissolution. Leachant renewal at the geometrical interface causes washing away of the surface of the waste form.

In porous wastes, which occur much more commonly, leaching is initiated at the pore scale, or the particle interface. These wastes consist of individual particles, which may or may not be consolidated, with voids between the particles.

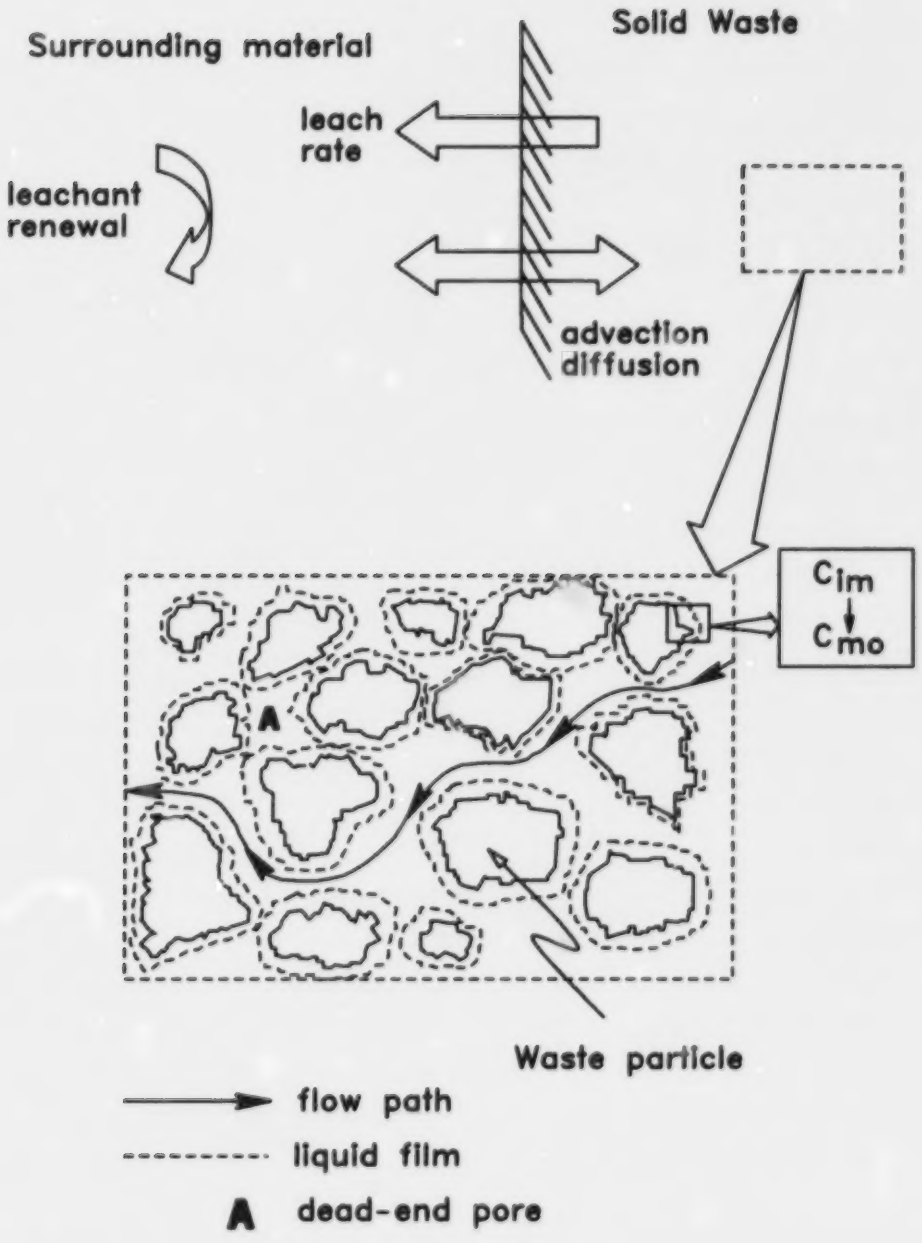


FIGURE 4 WASTE-LEACHANT INTERACTIONS

If a porous waste is wet, there could be many different phases present: several solid phases, an aqueous phase, a nonaqueous phase liquid, and a gas phase. Before contact with a leachant, the waste is normally at or approaching a state of chemical equilibrium, contaminants are associated with specific phases, and there is no net transfer between phases. Contacting the solid waste with leachant disrupts this chemical equilibrium, initiating the leaching process. The new system may evolve towards a new equilibrium state if sufficient time is available, provided that the leachant is not renewed.

For wastes that are initially dry, wetting initiates leaching by mobilizing those constituents that are easily dissolved or desorbed. At the pore scale (Figure 4), immobile constituents (C_{im}) become mobile (C_{mo}), as a result of complex combinations of chemical reactions, such as dissolution, desorption, and subsequent transport through the liquid film of immobile water that surrounds a particle of waste, by molecular diffusion.

The ultimate impact of leaching on the environment occurs when contaminants are transported away from the waste. If water flows within the solid waste, advective transport causes contaminants that have been mobilized by reactions at the pore scale to flow through the waste at various velocities, which are equal to or less than the average groundwater velocity. The pore-water velocities vary considerably in both magnitude and direction because the pore water must travel a sinuous pathway (tortuosity) within the available pore spaces, termed effective porosity, which are connected in a nonuniform manner. The velocity variations at this scale result in individual elements of water moving faster or slower than the average velocity, which causes spreading (i.e., dilution) of the contaminant concentration with increasing distance from the source. If there is no flowing water within the porous waste, soluble constituents may still be transported by molecular diffusion as a result of concentration gradients. Together, mechanical dispersion and diffusion constitute the lumped parameter known as hydrodynamic dispersion.

Leaching, therefore, is the result of chemical reactions at the scale of individual particles, which act to mobilize contaminants, coupled with transport processes that may be governed by advection, diffusion, or a combination of both, depending on the magnitude of leachant flow.

3 CLASSIFICATION OF LEACHING TESTS

For the purposes of this discussion, leaching tests have been separated into two broad categories on the basis of whether or not the leachant is renewed:

1) extraction tests (no leachant renewal)
2) dynamic tests (leachant renewal)

3.1 Extraction Tests

Extraction tests include all tests in which a specific amount of leachant is contacted with a specific amount of waste for a certain length of time, without leachant renewal. (This definition does not include analytical extractions or digestion procedures, which are used to measure the total contaminant concentration in a waste). The leachate is separated from the waste and analyzed either at various times during the test, to monitor the approach to a constant concentration and to derive kinetic information, or, as is done in most extraction tests, at the end of the test.

The underlying assumption in this type of test is that a steady-state condition is achieved by the end of the extraction test (i.e., the concentrations of constituents in the leachate become constant by the end of the test). In this no-flow system, a steady-state condition occurs when there is no net transfer of components from the liquid to the solid, or vice versa. Since the dissolution reactions in such complex systems are seldom reversible, this state is not the same as thermodynamic equilibrium.

Sampling in an extraction test over time to derive kinetic information or to monitor the attainment of a constant concentration is difficult since it must be done without modifying the waste-leachant interactions, which are a function of factors such as the liquid-to-solid ratio, and gaseous exchanges. This can be accomplished in three ways:

1) nondestructive sampling and analysis of parameters such as pH, conductivity, or specific ions
2) removing small volumes (aliquots) that are negligible when compared with the total volume
3) preparing as many extraction tests as data points required, and performing destructive analyses

Extraction tests can be further divided into four subcategories:

1) agitated extraction tests
2) nonagitated extraction tests

3) sequential chemical extraction tests
4) concentration buildup tests

3.1.1 Agitated Extraction Tests. Agitated extraction tests (Figure 5) are performed to reach steady-state conditions as quickly as possible. They measure the chemical properties of a waste-leachant system, as opposed to physical rate-limiting mechanisms. Agitation ensures a homogeneous mixture and promotes contact between the waste and the leachant. Sample particle size reduction is often performed to increase the surface area of contact and to eliminate mass-transfer limitations. This reduces the duration of the test by reducing the time required to reach a steady-state condition in the leachate. This procedure may also have the effect of overestimating the short-term release of constituents.

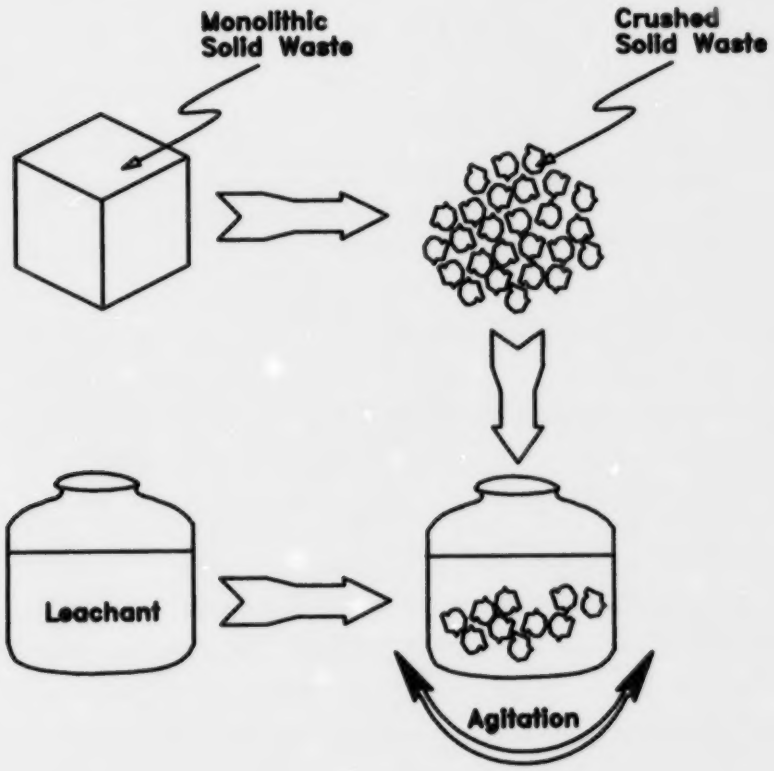
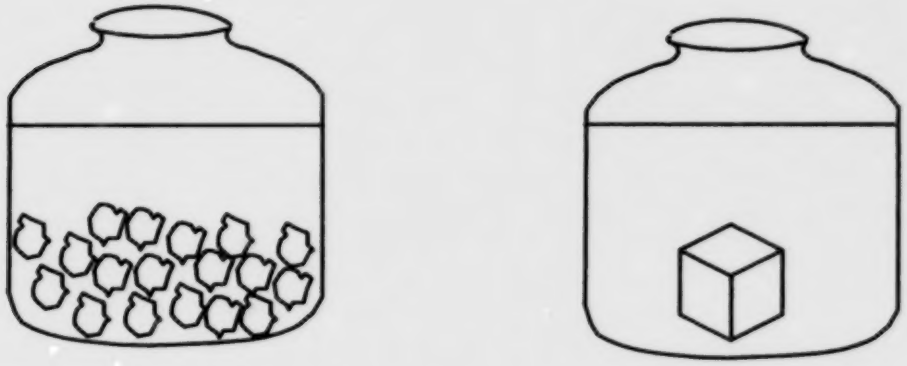


FIGURE 5 AGITATED EXTRACTION TEST

A steady-state leaching environment can also be attained in a column apparatus through recirculation of the leachate in a closed loop.

3.1.2 Nonagitated Extraction Tests. A nonagitated extraction test is performed to study the physical mechanisms that are rate limiting in leaching. The underlying assumption behind a nonagitated extraction test is that the physical integrity of the waste matrix affects the amount of contaminants that are leached during the test. Two types of nonagitated tests are illustrated in Figure 6. They can be performed on nonmonolithic samples (Figure 6a) or monolithic samples (Figure 6b).



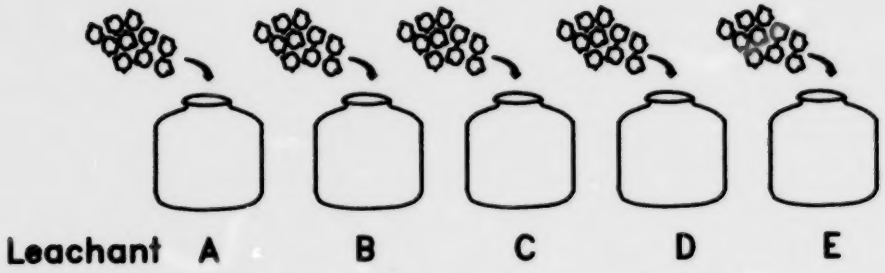
a) Static test with nonmonolithic solid waste

b) Static test with monolithic solid waste

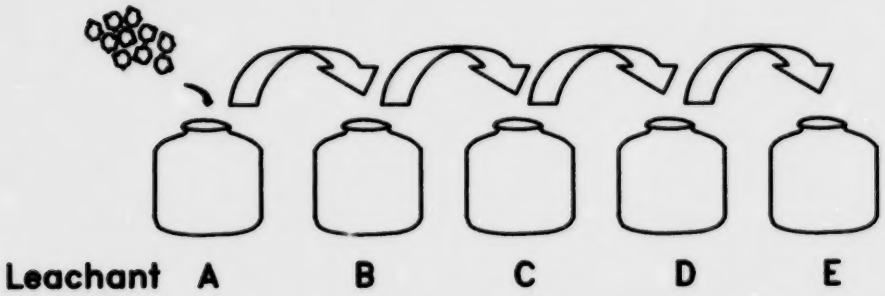
FIGURE 6 NONAGITATED EXTRACTION TESTS

The disadvantage of running a nonagitated test is that a much longer contact period may be required to reach steady-state conditions than required in an agitated test. The advantage of this type of test is that rate-limiting mechanisms of leaching, due to the physical integrity of the waste form, are taken into account.

3.1.3 Sequential Chemical Extraction Tests. A sequential chemical extraction test is composed of a battery of agitated extraction tests (Figure 7). It involves performing sequential elutions of aliquots of sample with different leachants (i.e., A, B, C, D, and E in Figure 7), which are increasingly more aggressive, in terms of chemical attack, towards the waste (Figure 7a). This method assumes that each successive leachant also extracts the sum of contaminants extracted by all preceding leachants. This test can also be conducted by subjecting the same aliquot of sample to each leachant (Figure 7b). The amount extracted in each elution is associated with a certain chemical form or mineral phase in the waste matrix. Although these amounts are operationally defined (e.g.,



a) With different waste samples



b) With the same waste sample and liquid/solid separation between elutions

FIGURE 7 SEQUENTIAL CHEMICAL EXTRACTION TESTS (with five leachants)

peroxide extractable), the fractions obtained are not very well defined in terms of chemical speciation.

3.1.4 Concentration Buildup Tests. In a concentration buildup test, an extraction is achieved at a very low cumulative liquid-to-solid ratio. Aliquots of samples of waste are successively contacted with the same leachant (Figure 8). The contact of leachate with fresh waste can be considered to model an elemental volume of water flowing through a large body of waste, approaching saturation with respect to specific contaminants. The purpose of this test is not to collect kinetic information, but to characterize a leachate saturated with soluble waste constituents. In some cases, this may simulate the actual pore water composition of a waste.

3.2 Dynamic Tests

Dynamic tests include all tests in which the leachant is continuously or intermittently renewed to maintain a driving force for leaching. The intermittent tests

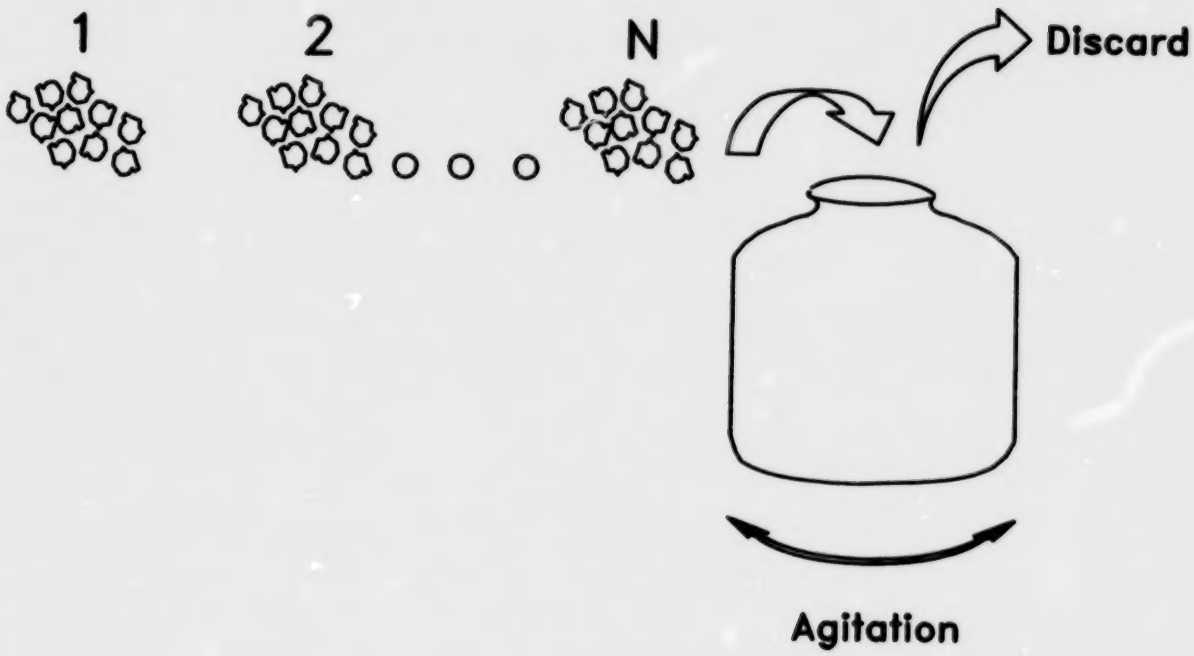


FIGURE 8 CONCENTRATION BUILDUP TEST (N waste samples)

may be conducted by alternating leaching periods with dry periods to study the effects of desiccation or unsaturated flow conditions.

Dynamic tests provide information about the kinetics of contaminant mobilization. Information is generated as a function of time, and attempts are often made to preserve the waste form's structural integrity. These two factors lend this category of leaching tests to the investigation of more complex mechanisms of leaching.

Dynamic tests can be further divided into four subcategories according to how the interface between the waste and the leachant is defined. Tests in which individual waste particles are used to define the interface are called serial batch tests. Tests in which a characteristic dimension of the waste, such as the external geometric surface area or the geometric surface area perpendicular to flow, is used to define the interface include

1) flow-around tests
2) flow-through tests
3) soxhlet tests

3.2.1 Serial Batch Tests. A serial batch test is conducted using a granular or crushed monolithic waste sample, which is mixed with leachant at a given liquid-to-solid ratio, for a specified period of time (Figure 9). The leachate is then separated from the solids, and replaced with fresh leachant until the desired number of leaching periods have been completed. The waste/leachant mixture is normally agitated to promote contact. Kinetic information regarding contaminant dissolution is obtained using the concentrations measured in the leachate from each of the leaching periods. Data from serial batch tests can be used to construct an extraction profile, which can be used to infer the temporal release of leachable constituents.

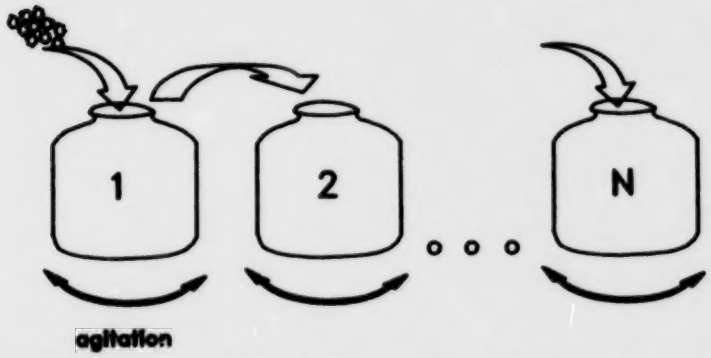
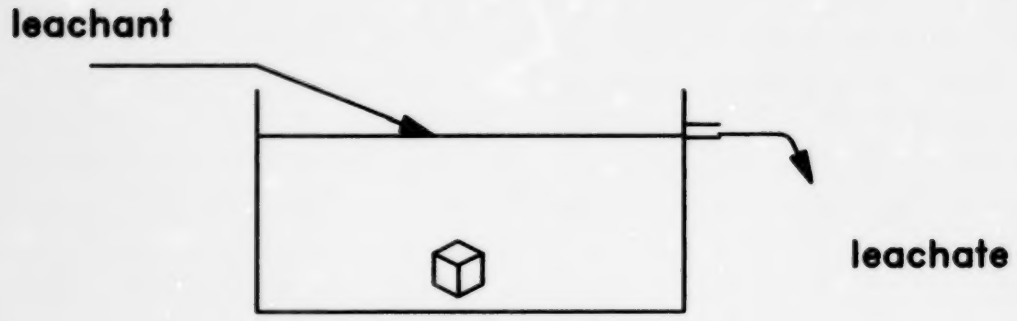


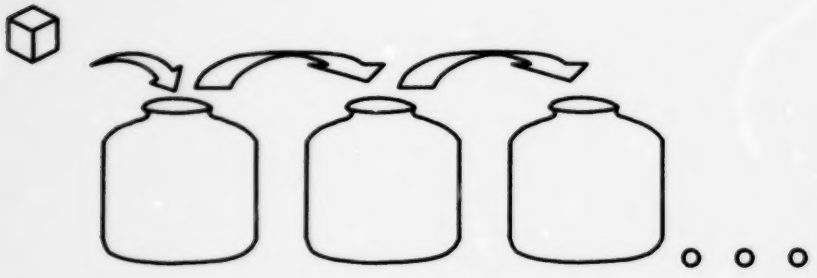
FIGURE 9 SERIAL BATCH TEST (N elutions)

3.2.2 Flow-around Tests. In flow-around tests, a sample of waste is placed in the leaching vessel and the flow of fresh leachant around the waste provides the driving force to maintain leaching. The liquid-to-solid ratio is expressed as the volume of leachant divided by the surface area of the solid sample. Samples are usually monolithic, although nonmonolithic or crushed waste may be used if it is confined in some manner. Agitation is generally not performed. Leachant flow is either continuous (Figure 10a), in which case it is sampled and analyzed periodically, or it is intermittently renewed (Figure 10b). The latter method is generally simpler from an experimental point of view, but the renewal frequency must be sufficient to prevent a buildup of contaminants at the waste/leachant interface, which may inhibit further leaching.

3.2.3 Flow-through Tests. In a flow-through test, an open container is packed with a porous solid through which leachant is passed, either continuously or intermittently. The effluent is sampled periodically and analyzed for the parameters of interest. The results are used to examine contaminant removal in which the primary transport mechanism is



a) Continuous leachant renewal



b) Intermittent leachant renewal

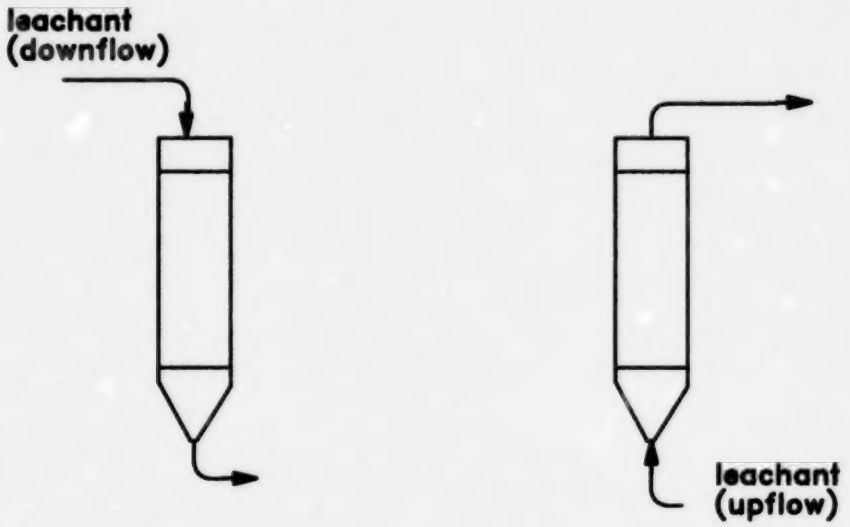
FIGURE 10 FLOW-AROUND TESTS

advection. There are two basic types of flow-through tests characterized primarily by the shape and size of the container:

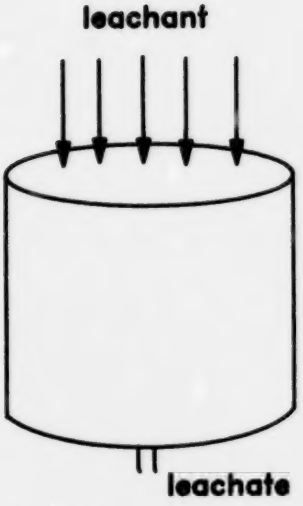
1) a column test is performed using a small cylindrical container (Figure 11a)
2) a lysimeter test is carried out in a large rectangular or cylindrical container (Figure 11b)

In general, the size of the sample used in a flow-through test tends to be large, to minimize the effects of sample heterogeneity and wall channelling effects. The depth of waste in either type of test varies according to the individual experiment.

Columns may be operated either in an upflow or downflow mode, whereas lysimeters are always operated in a downflow mode. Flow through the waste depends upon the hydraulic conductivity of the waste as well as the hydraulic gradient, and varies with the individual test.



a) Columns



b) Lysimeter

FIGURE 11 FLOW-THROUGH TESTS

Minicolumns[2] may be used to achieve a relatively rapid breakthrough of leached species. Since head losses may be large, and a rapid breakthrough is desired, the leachant is usually delivered under pressure, and at a constant flow rate. The advantages of minicolumns include 1) liquid-to-solid ratios that are similar to those of real waste-leachant systems, 2) a known and easily varied average fluid velocity, 3) negligible axial

dispersion or spreading of the solute, 4) a simple estimation of both equilibrium and kinetic coefficients, and 5) automation permitting the rapid output of data.

Care should be taken when conducting flow-through tests to avoid unnatural channelling of water and clogging by fine material or biological growth. In lysimeter tests, channelling cannot be avoided. It is a factor that occurs in the field, and its influence should be modelled in the laboratory. Biodegradation of organics can also be a problem in columns, although in some cases experiments are intentionally set up to measure the effects of biological activity. Flow-through tests can also be modified to examine other site-specific influences, such as vegetation on the surface of the container, or layered media, such as waste and geological material.

3.2.4 Soxhlet Tests. A soxhlet test can be used to continuously contact the waste sample with fresh leachant without adding or removing leachant from the apparatus (Figure 12). In a soxhlet test, the leachant is not renewed volumetrically. Rather, the

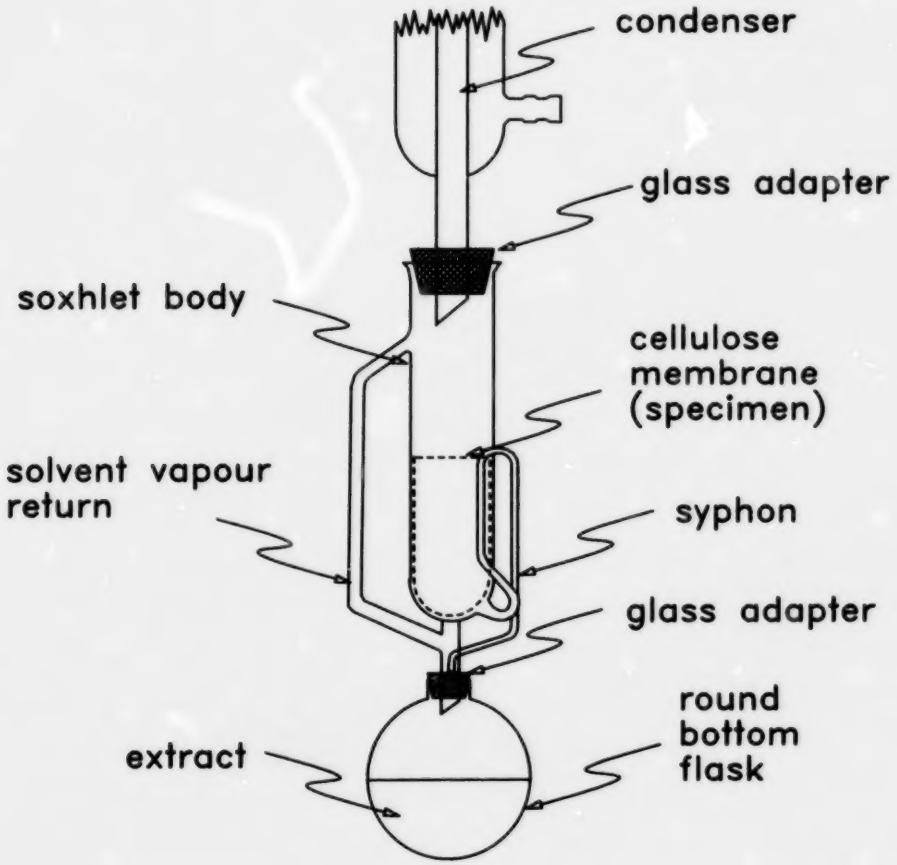


FIGURE 12 SOXHLET TEST

sample is continuously contacted with leachant, which has the leached constituents removed from it by evaporation and condensation prior to contact. The purpose of the test is not to collect kinetic information but to obtain the maximum amount of a constituent leachable from a waste sample, quickly and under severe conditions. In a soxhlet apparatus, the leachate is boiled, condensed, and recirculated repeatedly through or around the waste sample, depending upon its physical structure. A soxhlet test permits very high liquid-to-solid ratios and yet concentrates the leached constituents, avoiding analytical detection limitations. It is limited to using low boiling point liquids as leachants, and cannot be used to study chemical species that are volatile at the boiling temperature of the leachate.

4 MAIN LEACHING TEST VARIABLES

This chapter introduces the reader to seven experimental variables common to all extraction and dynamic tests:

- sample preparation
- leachant composition
- method of contact
- liquid-to-solid ratio
- contact time
- temperature
- leachate separation

This discussion should assist the reader in designing a leaching test to meet specific requirements, or in selecting a leaching test from among the protocols presented in Chapter 5.

4.1 Sample Preparation

Depending on the nature of the waste and the test to be performed, the sample may require one of the following preparatory steps:

- liquid/solid separation
- subsampling
- particle-size reduction
- surface washing
- compaction
- preservation
- curing
- aging

Liquid/solid separation may be performed on wastes containing a free liquid phase. The leaching test is then conducted on the solid portion of the waste only. The free liquid phase constitutes the initial leachate, which may be analyzed separately to estimate the pore-water concentration or included with the final leachate for analysis. Liquid/solid separation can be accomplished by various methods, including settling and decanting, centrifugation, and pressure filtration through filter media of various types.

Subsampling is required when several different tests or replicates are to be run on the same waste sample. Waste samples should be thoroughly mixed before subsampling is performed.

Particle-size reduction is required for most extraction tests. The goal is to reduce the time required to reach steady-state conditions by increasing the surface area of contact between the waste and the leachant. However, care should be taken to prevent the loss of volatile compounds in the leachate, if they are of interest. Particle-size reduction is usually carried out by grinding (e.g., mortar and pestle, or centrifugal grinder). To avoid contamination, which increases with increasing hardness of the material being ground, tungsten carbide should be selected as a grinder material.

Surface washing may be performed prior to testing small monolithic samples in flow-around tests. The surface is washed to remove small detachable particles and soluble salts by quickly dipping the sample in an aqueous solution.

Compaction or remolding is often required for flow-through tests. Reproducibility and field simulation considerations require that samples be compacted to a prespecified density, using methods such as vibration, proctor compaction, or modified proctor compaction.

Sample preservation is performed to avoid biological activity. This is a greater problem in tests of long duration, such as column tests. Various chemical treatments are available, such as sodium azide; however, none offer complete efficacy.

Curing may be performed on waste samples that have been transformed into a solidified mass by various chemical additives, such as portland cement. It allows the waste sample to gain physical and engineering properties, such as high unconfined compressive strength and low permeability, that are considered to be important in reducing leachability. Curing can be used to achieve a variety of chemical reactions within the waste, although this term usually refers to cement hydration.

Aging may be performed on any type of waste sample to account for the physical, chemical, and biological alterations that a waste might undergo in the field. An example of aging would be oxic incubation of sediments dredged from an anoxic environment, prior to performing a leaching test.

4.2 Leachant Composition

The release of contaminants from a waste in any leaching test may be strongly influenced by the leachant composition, especially at high liquid-to-solid ratios, or with the use of an aggressive solution. Chemical properties of the leachant that influence contaminant mobilization are indicated in Table 3. Examples of three types of commonly used leachants, i.e., water, site liquid, and chemical solution, are identified in Table 4. Several advantages and disadvantages of these leachants are outlined in Table 5.

TABLE 3 IMPORTANT FACTORS OF LEACHANT COMPOSITION

Factor	Release Mechanism Affected
pH	Dissolution/precipitation of metals, speciation of inorganic species Adsorption/desorption of organics
Eh, redox potential	Oxidation/reduction of inorganic species
Ionic strength	Ionic exchange of metals, speciation chemistry, and solubility products
Chelating and complexing agents	Metal solubility
Buffering capacity	All above properties

TABLE 4 COMMONLY USED LEACHANTS

Type of Leachant	Common Uses	Examples
Water	Nonaggressive, baseline medium without buffering capacity	Distilled, deionized,[3] and tap water
Site liquid (real or synthetic)	Simulates site-specific leaching conditions	Rainwater,[4] groundwater,[5] surface water, landfill leachant,[6] seawater
Chemical solution	Examines metal speciation and organic compound binding	Strong chemical solution (acidic, basic, reducing, oxidizing, complexing, solvent, etc.)

4.3 Method of Contact

Since a leaching test is primarily a system to study the transfer of contaminants from a waste to a liquid, it is important to

1) consider the aspects of the test conditions, such as agitation, that promote mass transfer
2) take into account the effect of mass exchange with other components of the system, primarily the leaching vessel and the atmosphere

TABLE 5 ADVANTAGES AND DISADVANTAGES OF COMMONLY USED LEACHANTS

Leachant	Advantages	Disadvantages
Pure water	Reliable, simple standard Waste establishes the chemical environment	Lack of background composition may result in dissolution of common ions
Site liquid	Best field case model Several synthetic liquids available	Requires characterization (to obtain leaching results by subtraction) Results not comparable with other leaching studies Labour intensive (sampling and preservation)
Chemical solution	Allows for the study of waste chemistry	Aggressive, difficult to relate data to field conditions

Agitation of the leachant-waste mixture allows steady-state conditions to be reached at a faster rate by maintaining maximum contaminant concentration gradients at the leachant - waste particle interface. Different methods can be used to agitate the waste, including

- shaking (wrist action or reciprocation)
- stirring (magnetic or paddle)
- tumbling
- gas bubbling

In static or nonagitated tests, the leachant-waste interface is usually the geometrical surface area of the waste form. There is usually no provision for mixing because diffusion of leached constituents within the leachate is assumed to be much faster than the rate of release by mechanisms such as dissolution from the surface or diffusion from within the waste. It is important, however, to ensure that the leachate is well mixed before sampling.

It may be important to identify and quantify exchanges of chemical species other than between the waste and the leachant. Exchanges between the leachant and the leaching vessel are always undesirable, whereas exchanges with the atmosphere depend in

large part upon the objectives of the test.

To minimize exchanges with the leaching vessel, glass or stainless steel should be used for organic contaminants, and plastic for inorganic contaminants. If the cost is not prohibitive, polytetrafluoroethylene is considered to be acceptable for both. For the purpose of verifying the mass of constituents adsorbed to the container wall, the emptied leaching vessel can be extracted with a strong solvent.

The test system may be either open or closed to the atmosphere. The choice depends on the specific leaching problem being examined. For example, a closed system provides a better simulation of the saturated groundwater environment, whereas an open system models problems like acid mine drainage and unsaturated disposal environments more accurately.

An open system facilitates sampling, leachant renewal, and periodic or continuous adjustment of the pH or redox potential. However, a system that is open to the atmosphere allows for the loss of volatile compounds, including water and organics, and the introduction of CO_2 and O_2 from the air. Losses due to evaporation may have to be accounted for in an open system.

Several apparatus configurations will prevent volatile contaminants from escaping. If there is no headspace in the leaching vessel, volatiles will remain in either the solid phase or the leachate. If there is a headspace, volatiles will be partitioned in the gas phase. Analysis of the headspace allows for an evaluation of this loss.

Even for experiments carried out in closed containers and under controlled conditions, penetration of gases through plastic container walls can have a significant effect, especially over long durations.

4.4 Liquid-to-solid Ratio

The liquid-to-solid ratio is the ratio of the amount of leachant in contact with the waste to the amount of waste being leached. Although this definition appears straightforward at first glance, it can become confusing because of the many ways in which the two variables in the ratio can be defined.

The liquid-to-solid ratio can be expressed as

1) volume of leachant/mass of waste
2) mass of leachant/mass of waste
3) volume of leachant/surface area of waste (for monolithic wastes)

Furthermore, when using expressions 1 and 2, the mass of waste being

leached can be calculated on a wet weight or a dry weight basis. Another problem arises because of the various ways that the volume or mass of leachant can be calculated, depending on whether or not the liquid phase of the waste is included in the total leachant volume.

Figure 13 illustrates how these various ways of defining the amounts of waste and leachant can give different liquid-to-solid ratios for the same system. The three fractions shown in Figure 13 include the amount of leachant added, the liquid phase of the waste, and the solid phase of the waste.

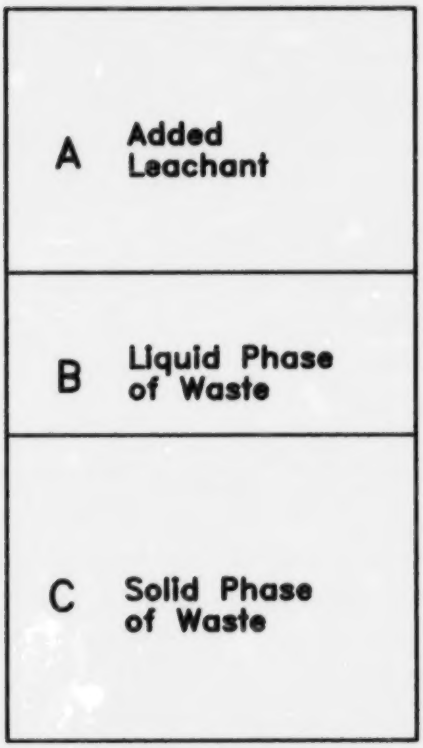


FIGURE 13 LIQUID AND SOLID FRACTIONS OF THE WASTE-LEACHANT SYSTEM

If the waste is dry, the liquid-to-solid ratio is simply A/C. If the waste is wet, however, there are three ways to define the liquid-to-solid ratio:

1) the waste is the sum of the liquid and solid phases, i.e., liquid-to-solid ratio = A/(B+C)
2) the leachant is the sum of the amount of leachant added plus the liquid phase of the waste, i.e., liquid-to-solid ratio = (A+B)/C

3) the liquid phase of the waste is excluded from the calculations, and waste is the solid phase only, i.e., liquid-to-solid ratio = A/C

The leachate concentration of highly soluble species (e.g., sodium, potassium) is inversely proportional to the liquid-to-solid ratio if all of the species have been removed from the waste. However, if the release of a species is limited by solubility, the final concentration is independent of the liquid-to-solid ratio and simply equals the maximum solubility concentration. In general, the leachate concentration will be controlled by a number of competing factors: the amount of contaminant available, solubility, and kinetically controlled chemical reactions. Thus, the relationship between the liquid-to-solid ratio and concentration is complex and different for each species of interest.

Selection of an appropriate liquid-to-solid ratio depends on the objectives of the leaching test, the solubility of species of interest, and analytical constraints. The ratio should be low enough to avoid dilution of contaminants to less than analytical detection limits. On the other hand, however, the ratio must be high enough to prevent solubility constraints from limiting the amount of contaminants that can be leached from the waste. The selected ratio should be somewhere between these two limitations. Practical values for the liquid-to-solid ratio range from 1:1 to 100:1.

4.5 Contact Time

The total amount of time that a leachant is in contact with a waste sample before the attainment of steady-state conditions will influence the amount of contaminant released. In extraction tests, the contact time is equivalent to the duration of the test; whereas in dynamic tests, it is a function of the flow rate, or the number of elutions, in addition to the test duration.

The contact time for extraction tests should allow steady-state conditions to be reached for the contaminants of interest. This is generally on the order of hours to days for samples that have undergone particle-size reduction. For monolithic samples, it can be on the order of weeks to months.

The contact time for dynamic tests should be sufficient to allow for observation of the processes of interest. Diffusion processes may be quantified within a few weeks, although several months may be required to study slow chemical reactions.

4.6 Temperature

Temperature affects the results of extraction and dynamic tests. Both the van't Hoff relationship, which applies to thermodynamic equilibrium constants and

solubility products, and the Arrhenius relationship, which applies to kinetic processes such as desorption and diffusion, indicate that properties or mechanisms relevant to leaching vary exponentially with temperature.

For convenience, most leaching tests are performed at room temperature. Higher temperatures may be used to accelerate the rate of leaching, although this may also change the properties of the waste, or to simulate the effects of biological activity in a landfill or the self-heating of radioactive wastes. Table 6 gives an indication of the average temperatures encountered in various disposal environments.

TABLE 6 AVERAGE TEMPERATURES OF DIFFERENT DISPOSAL ENVIRONMENTS

Disposal Scenario	Temperature Range
Surface impoundment	Ambient (i.e., above and below freezing)
Municipal solid waste landfill/codisposal	10°C to 45°C
Deep-well, hydrofracture injection	8°C to 15°C
Hazardous waste landfill, monofill	Above freezing (after closure)
Utilization	Ambient

4.7 Leachate Separation

Leachates are commonly separated from agitated nonmonolithic wastes by filtration using a 0.45 μm membrane filter (a convention used to define soluble species). However, very small particles called colloids are known to move with advective groundwater flow and may carry adsorbed contaminants. A smaller pore size filter may be used if these particles are to be removed.

Glass fibre filters are chosen when hydrophobic, low solubility organic molecules are expected in the leachate since they may have a high affinity for filters composed of an organic polymer. Membrane filters, such as cellulose acetate, should be used for metal species in place of glass. The same care used to select a leaching vessel should be applied when selecting the filter material.

It may not be necessary to filter the leachate from nonagitated monolithic samples if the method of contact generates only dissolved species. This should be verified before sampling.

5 LEACHING TEST PROTOCOLS

This chapter presents available leaching test protocols. Its structure parallels that of Chapter 3, in which tests were classified and described in general terms. Here, a brief history of each protocol, the intended purpose of the protocol, and any noteworthy features of the method are presented. Summaries of the test variables and references to the method are presented in tabular form. Additional supporting references for each type of test are listed in the bibliography.

Protocols selected for presentation satisfied one or more of the following criteria:

- the protocol is sponsored by a recognized organization
- it is a voluntary or regulatory standard
- it is a publicly available protocol
- representative results are published in the literature

5.1 Extraction Tests

5.1.1 Agitated Extraction Tests (Table 7).

EP Tox (EPA Method 1310). The Extraction Procedure Toxicity Test (EP Tox) was promulgated in 1980, under the Resource Conservation and Recovery Act (RCRA), to classify wastes as hazardous or nonhazardous, based on maximum permissible concentrations for eight metals, four pesticides, and two herbicides. This test was based upon a 95% municipal/5% industrial codisposal mismanagement scenario, primarily for inorganic wastes. It requires structural integrity testing for monolithic waste samples, and intermittent pH adjustment.

Promulgation of EP Tox by the United States Environmental Protection Agency (U.S. EPA) has spawned the development of a whole family of similar protocols, including the Toxicity Characteristics Leaching Procedure (TCLP), the Ontario Reg 309 Leachate Extraction Procedure (LEP), the Quebec R.s.Q. (Q.R.s.Q.), and the California Waste Extraction Test (WET).

LEP. The Leachate Extraction Procedure (LEP) is the regulatory extraction test used in the province of Ontario, Canada. It was promulgated under Regulation 309 of the Ontario Environmental Protection Act in 1985. Only minor changes to EP Tox were made in developing this test. The Provisional Standard for Leachate Extraction Procedure of the Canadian General Standards Board (CGSB) is identical to LEP and has been adopted by the

TABLE 7 AGITATED EXTRACTION TESTS

Test Name and Proponent	Status of Development	Leaching Vessel	Sample Preparation	Sample Mass	Leachant	Liquid-to-solid Ratio	Agitation	Duration	Leachate Separation
EP Tox[7] U.S. EPA Method 1310	Standard regulatory method (1980)	Unspecified	Nonmonolithic waste: phase separation Monolithic waste: particle-size reduction	100 g	Deionized water 0.5 N acetic acid (max. 2.0 meq H^+/g solid)	20:1	Unspecified, continuous	24 to 28 hours	0.45 µm filtration
LEP[8] MOE (Ontario)	Standard regulatory method (1985)	Wide mouth, 1250 mL cylindrical bottle	Phase separation by 0.45 µm membrane filter	50 g of dry solids	Distilled water Acetic acid (2.0 meq H^+/g dry solids)	20:1	End over end (10 rpm)	24 hours	0.45 µm filtration
TCLP[9] U.S. EPA Method 1311	Standard method (1986)	Any material compatible with waste, zero headspace extractor (ZHE) for volatiles	Cutting/crushing and grinding Solid/liquid phase separation No structural integrity	100 g (25 g for ZHE)	Buffered acetic acid 1) pH = 4.93 2) pH = 2.88	20:1	End over end (30 rpm)	18 hours	0.6 to 0.8 µm borosilicate glass fibre filter combines liquid phase with extract
Q.R.s.Q.[10] MOE (Quebec)	Standard regulatory method (1987)	>1 L bottle	No phase separation Grinding No structural integrity	100 g dry solids 50 g for volatiles	Inorganics: buffered acetic acid (0.82 meq H^+/g dry solids) Organics: distilled water	10:1	End over end (10 to 20 rpm)	24 hours	30 min decantation, 0.45 µm filtration
WET[11] California	Standard regulatory method (1985)	Polyethylene or glass container	Milling, 0.45 µm filtration	50 g	0.2 M sodium citrate at pH 5.0	10:1	Table shaker Rotary Extractor	48 hours	Centrifugation, 0.45 µm filtration
French Leach Test[12] AFNOR (France)	Proposed standard for hazardous waste (1987)	Straight wall, 1.5 L bottle	Remove free liquid Reduce particle size to <9.5 mm	100 g	Demineralized water	10:1	Roller or shaker	16 hours	0.45 µm filtration
EE[13] Environment Canada	Published research method	Inorganic: wide mouth, plastic sample bottle (250 mL) Organic: glass (500 mL)	Grinding (inorganic) Mortar and pestle (organic)	Inorganics: 40 g Organics: 80 g	Distilled water	4:1	National Bureau of Standards rotary extractor	7 days	0.45 µm vacuum screen
ASTM D3987[14] ASTM	Standard research method	Round, wide mouth bottle	As received	700 g	Distilled water (ASTM Type IV)	4:1	Shaking	48 hours	0.45 µm filtration
MBLP[15] Environment Canada	Published research method	Square, polyethylene or glass bottle, 1 to 2 L	Remove free liquid Reduce particle size to <9.5 mm	Variable to fill 90% of bottle	Distilled water Acidic water buffer to pH 4.5 Synthetic municipal solid waste	4:1 or 2:1	Slow rotary tumbling	24 hours	0.45 µm filtration
MCC-3S[16] Materials Characterization Center	Standard regulatory method (radioactive wastes)	Teflon container, 20 mL to 1 L	Crush waste form into two fractions: 74 to 149 µm 180 to 425 µm	>1 g	Choice of high purity water, silicate water, brine, repository water	10:1	Rolling and rocking	Variable: 28 days to several years	N/A

Note: AFNOR = Agence Française de Normalisation

provinces of British Columbia, Alberta, and Manitoba. The minor variations in methodology between LEP and EP Tox result in only one practical difference: in the case of limed wastes with high moisture content and little free liquid, the pH of the final extract is higher using LEP, which results in less dissolution of some metals.

TCLP (EPA Method 1311). The Toxicity Characteristics Leaching Procedure (TCLP) was developed in 1984 under the Hazardous and Solid Waste Amendments to RCRA. It was promulgated in 1986 under the Hazardous Waste Management System Land Disposal Restrictions, and U.S. EPA plans to promulgate it for use in hazard determination to replace EP Tox. TCLP was based on the same assumptions as EP Tox, but it includes the following modifications. Volatiles are prevented from escaping to the atmosphere by using a modified leaching vessel called a zero headspace extractor (ZHE), which eliminates the headspace. Two leachants are employed in the procedure. For highly alkaline wastes, a solution of acetic acid is used (pH = 2.88); whereas for other wastes, use of a buffered leachant (pH = 4.93) eliminates the need for continual pH adjustment. In either case, the maximum amount of acid addition is the same, i.e., 2 meq/g waste. There is no allowance for structural integrity testing of monolithic samples, and all wastes must be ground to a particle size of less than 9.5 mm. Compared with EP Tox, TCLP increases by an order of magnitude the number of organics analyzed. Other equipment changes and specifications were made to improve reproducibility and reduce contamination. All of these changes have resulted in only slightly greater metal concentrations in TCLP leachates compared with those in EP Tox leachates.

EPA Method 1312, a variant of Method 1311, employs simulated acid rain and was developed in 1988 by U.S. EPA for use in assessing the impact that contaminated soils may have on groundwater.

Quebec R.s.Q., Q-2, R 12.1 (Q.R.s.Q.). Q.R.s.Q. is the regulatory test used in the province of Quebec, Canada. It was first developed in 1980, and promulgated in 1985, with no provision for organics. In 1987, it was included in the Procedure for Evaluating the Physical and Chemical Characteristics of Solid and Liquid Wastes. Both EP Tox and TCLP were referred to in its development, with the resulting test being very similar to the latter. In Q.R.s.Q., however, phase separation is not performed, and the liquid part of the waste becomes part of the extracting fluid or leachant. Also, only distilled water is used as a leachant for organics.

California Waste Extraction Test (WET). First submitted in 1984, WET forms part of an overall legislation to identify hazardous and extremely hazardous wastes. WET was

established to determine the amount of extractable substances in a waste that have been identified as being hazardous according to the Persistent and Bioaccumulative Toxic Substances criteria. This test is very similar to EP Tox; however, the addition of sodium citrate in the leachant as a chelating agent may make this test more aggressive towards certain wastes, or waste components.

French Leach Test. This French protocol is a standard regulatory leach test used to determine the soluble fraction of a solid waste in an aqueous solution, under strictly defined test conditions. The test cannot be used to analyze environmental impact or treatment technologies, such as solidification. It is intended for solids, pastes, and particles.

ASTM D3987. D3987 was standardized by the American Society for Testing and Materials (ASTM) in 1981, and a revised version was published in 1985. It still stands as a basic single batch agitated extraction test; however, many modifications have since been proposed or developed. Its intent is to provide a rapid, standard extraction procedure for industry. It is not intended to simulate site-specific conditions. The final pH of the leachate reflects the interaction of the leachant with the buffering capacity of the waste. It has not yet been validated for organic wastes.

EE. The Equilibrium Extraction (EE) protocol was first published in 1986 by Environment Canada. It was based upon ASTM D3987. EE uses distilled water and a low liquid-to-solid ratio to let the waste establish the chemical environment. Also, a long test duration (7 days) and particle-size reduction (<100 mesh) increase the probability of achieving steady-state conditions in the leachate. The procedure is run separately for organic and inorganic wastes, using appropriate containers and crushing methods.

MBLP. The Multiple Batch Leaching Procedure (MBLP) was based on experimentation carried out at Environment Canada's Wastewater Technology Centre between 1980 and 1986. This battery of tests allows for comparisons of the leachability of various wastes by measuring the change in leachability under different test conditions. Single elutions are performed, but under a variety of test conditions: two liquid-to-solid ratios, and three leachants. Two of the extractions are similar to EP Tox, the difference being that less acid is added and there is no phase separation.

MCC-3. The agitated powder extraction test (MCC-3) of the Materials Characterization Center (MCC) is one in a series of test methods that were developed to help evaluate the chemical durability of nuclear waste forms. This test determines the maximum

concentration of elements in solution under steady-state conditions. It is applied to crushed samples of monolithic waste forms. This test is similar in intent to that of MCC-1 and MCC-2 in which monolithic samples are tested, but the increased surface area exposed to the leachant makes it a more rapid test. To derive kinetic information, a separate powdered specimen, leach container, and leachant are required for each data point.

5.1.2 Nonagitated Extraction Tests (Table 8).

MCC-1, MCC-2. These two in the series of Materials Characterization Center (MCC) tests were developed to compare the leaching behaviour of waste forms of varying formulation. They were designed for both a rapid evaluation and for gaining a

TABLE 8 NONAGITATED EXTRACTION TEST PROTOCOLS

Test name:	MCC-1[17] Static Leach Test Method MCC-2[18] High Temperature Static Leach Test Method
Proponent:	Materials Characterization Center
Status of development:	Standard method for evaluation of nuclear waste forms
Leaching vessel:	Teflon, 20 mL to 1 L, environmental chamber (30°C (MCC-1) to 100°C (MCC-2))
Sample preparation:	1) Test material characterization (optical microscopy, X-ray diffraction, scanning electron microscopy - X-ray emission (SEM-XRE), bulk chemical, radiochemical analysis, autoradiography) 2) Specimen fabrication or cutting 3) Ultrasonic surface washing
Sample mass:	Recommended surface area = 40 mm^2
Leachant:	Three standard leachants: • water • brine • silicate/bicarbonate Also, site-specific leachants
Volume/surface area:	10 cm
Duration:	7, 28, or 364 days, or long-term options
Leachate separation:	0.45 μm filtration

comprehensive understanding of a waste form's long-term behaviour. It is not possible, however, to make predictions of in situ performance from these tests. They apply to monolithic, inorganic, high level radioactive waste forms, including glasses, cements, and crystalline ceramics. MCC-1 is conducted at temperatures below 100°C, whereas MCC-2 is performed above this temperature. The method has been divided into a series of options that become increasingly more complex by changing the temperature, test duration, and sampling frequency.

5.1.3 Sequential Chemical Extraction Tests (Table 9).

SCE. The Sequential Chemical Extraction (SCE) test method was developed to examine the partitioning of specific trace metals into different fractions or chemical forms. This test implies that all forms of a given metal do not have an equal impact on the environment, and it simulates, to a certain extent, the range of environmental conditions to which a waste may be subjected in the field. Various sequences of chemical leachants are used to cause a change in the chemical environment, and subsequent release of a specific fraction. The data cannot be translated directly to the field since they give an indication of gross change, but are not quantitative. A similar test has yet to be developed to study the binding strength of organic contaminants on waste matrices.

TABLE 9 SEQUENTIAL CHEMICAL EXTRACTION[13] TEST PROTOCOL

Fraction	Target Metal	Release Subject to Changes in	Leachant
A	Ion exchangeable	Ionic composition	0.25 M CaCl 0.75 M LiCl 60% CH_3OH
B	Surface oxide and carbonate bound	pH	1 M CH_3COONa in CH_3COOH; pH = 5
C	Bound to Fe and Mn oxides	Eh	1 M $NH_2OH{\cdot}HCl$ in 25% CH_3COOH
D	Bound to organics	Eh	0.02 M HNO_3 in 30% H_2O_2 1.2 M CH_3COONH_4 in 20% HNO_3
E	Residual	Stable	30% H_2O_2, HF, HCl

5.1.4 Concentration Buildup Tests (Table 10).

SLT (Procedure C, University of Wisconsin). Sponsored by U.S. EPA, the Standard Leaching Test (SLT) was developed at the University of Wisconsin in 1978, in one of the early attempts to establish a standard test to evaluate the relative leaching potential of a waste. This test differs significantly from standard extractions in that the filtered waste material is discarded after each elution, and the leachate is kept and then mixed with a fresh sample.

TABLE 10 CONCENTRATION BUILDUP TEST PROTOCOL

Test name:	Standard Leach Test[19] (Procedure C)
Proponent:	University of Wisconsin, U.S. EPA
Status of development:	Past standard
Leaching vessel:	Square flasks
Sample preparation:	Liquid separation, 0.45 µm pressure filtration
Sample mass:	120 g (wet weight)
Leachant:	Distilled water, synthetic municipal landfill leachate
Liquid-to-solid ratio:	10:1, 7.5:1, 5:1 (dry weight)
Agitation:	Rotating shaker
Elution time:	24 hours
Number of elutions:	3
Leachate separation:	0.45 µm filtration
Test duration:	3 or 14 days

5.2 Dynamic Tests

5.2.1 Serial Batch Tests (Table 11).

MEP (EPA Method 1320). U.S. EPA's Multiple Extraction Procedure (MEP) is based entirely on EP Tox. It was designed to simulate the leaching that a waste would undergo if it were exposed to repeated events of acid precipitation in an improperly designed

TABLE 11 SERIAL BATCH TESTS

Test Name and Proponent	Status of Development	Sample Preparation	Leachant	Leaching Vessel	Agitation	Sample Mass	Liquid-to-solid Ratio	Contact Time	Number of Elutions	Leachate Separation
MEP(20) U.S. EPA Method 1320	Standard test method (1986)	Same as EP Tox	Acetic acid Synthetic acid Distilled water	Same as EP Tox	Same as EP Tox	Same as EP Tox	Same as EP Tox	Same as EP Tox	10	Same as EP Tox
MWEP(21) U.S. EPA	Technical resource document (1986)	Particle-size reduction to <9.5 mm or structural integrity	Distilled water Site water	Wide mouth sample bottle	Rotary tumbler	Unspecified	10:1	18 hours	4	Settling and filtration
Graded Serial Batch(22) U.S. Army	Research method for waste and soil (1987)	-	Distilled water	Unspecified	Periodic gentle shaking (4/5 times daily)	300 g	2/3/6/12/24/48/96:1	Until steady-state conditions attained	>7	Vacuum filtration
SBE(23) D4793-88 ASTM	Standard method (proposed) (1988)	Drying Phase separation	Reagent water (Type II D1193)	2 L, wide mouth bottle	None	100 g	20:1	24 hours	10	0.45 µm membrane filter
WRU Leach Test(24) Harwell Laboratory United Kingdom	Standard method (1982)	Crushing Vacuum filtration	Distilled water Dilute acetic acid buffered (pH = 5)	50 mL, wide necked flask	Mechanical flask shaker	100 g	One bed volume (first five elutions) 10 bed volumes (more than six elutions)	2 to 80 hours Steady state	5	Vacuum filtration
SLT Cascade Test(25) SOSUV Netherlands	Standard research method for combustion residues (1984)	Crushing/ seiving Dry	Distilled water Nitric acid (pH = 4.0)	1 L polyethylene bottle	Shake/roll	40 g	20:1	23 hours	5	Settling and 0.45 µm filtration

landfill. It determines the maximum leachate concentration under acidic conditions for solid, liquid, or multiphase wastes. It is conducted entirely according to the EP Tox protocol, except that nine successive elutions are performed on the same sample with a synthetic acid rain after initially conducting a distilled water elution.

MWEP. The Monofill Waste Extraction Procedure (MWEP) is a technical resource document (TRD) of U.S. EPA. It is to be used by writers and reviewers of permit applications for hazardous waste land disposal facilities. TRDs provide information on technologies and evaluation techniques, but they are not regulations in themselves. This test is intended to derive reasonable leachate compositions for industrial wastes subjected to monofilling in properly engineered facilities. It gives an indication of which constituents are potentially leachable, as well as the expected relative delay in their release, and it determines the maximum release under mildly acidic conditions. It does not attempt to simulate field leaching. MWEP was previously called the Solid Waste Leaching Procedure (SWLP). The MWEP protocol involves a four-step sequential batch extraction at 18 hours per elution, at a liquid-to-solid ratio of 10:1. It has been tested for a wide range of contaminants, except volatile organics. Although there is no provision for particle-size reduction, it has been observed that the rotary tumbler tends to crush many monolithic samples.

Graded Serial Batch. The Graded Serial Batch test was developed by the United States Army Corps of Engineers. It is a rapid and versatile research test that can be used to measure the leachability of selected wastes, the attenuation capacity of certain soils, and the effectiveness of various fixation processes for industrial wastes. It has been developed with the remediation of contaminated sites in mind, where more than one kind of waste and more than one kind of soil are present. The procedure involves repeated extractions of a waste, as well as sequential equilibrations of the waste extract with a soil. The same waste sample is extracted with increasing volumes of leachant, and the leachate is equilibrated in succession with the same three soil samples. This procedure can be repeated for any number of extractions.

ASTM D4793-88, SBE. The Sequential Batch Extraction (SBE) of ASTM is a modification of Method D3987 that allows for filtration and separation of the waste sample after the first distilled water elution, and subsequent extractions of the same waste, up to nine times.

WRU Leaching Test. The Waste Research Unit (WRU) at Harwell Laboratory in the United Kingdom has developed a serial batch method, called the repetitive shaker test, to

provide a simple method of quantifying the initial leaching of a waste to help assess any limitations on landfill disposal that should be imposed. It expresses the liquid-to-solid ratio in terms of a bed volume, which is the volume of leachant required to just saturate a waste. The protocol calls for first performing an equilibrium shaker test, which is a single elution agitated extraction, with hourly sampling to determine steady-state conditions. A repetitive shaker test is then conducted, which involves five serial batch extractions with one bed volume of leachant and a sixth extraction with 10 bed volumes of leachant (to represent the average leaching over 6 to 15 bed volumes).

SLT, Cascade Test. In 1981, the Netherlands recognized the need to develop standardized leaching tests for coal and municipal solid waste (MSW) incinerator ashes. In 1984, a series of standard leaching tests were published with the goal of closely approximating field leaching conditions, to improve the capability to predict the possible environmental effects of ash disposal. The Standard Leaching Test (SLT) in full involves evaluating the total composition of the residue, its behaviour in a column (the composition of the first leachate and the time to peak in a column test), its medium- and long-term leaching behaviour (cascade test), and its maximum leachability (a two-step extraction). The user decides among the various tests that are offered on a decision tree, based on the liquid-to-solid ratio that best approximates the time of leaching and amount of liquid that the waste would encounter in the field. The cascade test is a serial batch extraction with five elutions in which fresh acidified leachant is added at a liquid-to-solid ratio of 20:1, for a cumulative liquid-to-solid ratio of 100:1.

5.2.2 Flow-around Tests (Table 12).

MCC-4S. This test is intended to provide a data base on the leaching of nuclear waste forms under dynamic conditions, to establish a basis of comparing inorganic monoliths, and assist in repository licensing. It is based on a test developed at the Lawrence Livermore National Laboratory, and was modified to use commercially available equipment that can be operated at 100°C. The test parameters have been selected so that straightforward comparisons with other MCC tests can be made, the following of which have been standardized: leachant composition, temperature, flow rate, and volume-to-surface area ratio. Specimens are immersed in the leachant under single-pass, continuous-flow conditions. This involves using peristaltic pumps, reservoirs, and receiver bottles, in addition to an environmental chamber and leaching containers modified for high temperatures and radiation. Although the pumps are intended to control the flow rate to within ±10%, this is currently not attainable for the low-flow condition of 0.001

TABLE 12 FLOW-AROUND TESTS

Test Name and Proponent	Status of Development	Sample Preparation	Sample Size	Leachant	Leaching Vessel	Volume : Surface Area	Leachant Renewal Rate
MCC-4S(26) MCC	Standard (1983)	Individual fabrication or surface washing or cut sample	400 mm^2	Pure water, bicarbonate-silicate water, repository water	1 L teflon cylinder	10 cm	Flow rate = 0.1, 0.01, and 0.001 mL/min
IAEA Dynamic Leach Test(27) IAEA	Past standard (1971)	One circular face is prepared for leaching	Cylinder, 5 cm x 5 cm	Demineralized water Site water	Compatible with sample and leachant	<10 cm	Daily (1st week), weekly (8 weeks), monthly (6 months), biannually
ISO Leach Test(28) ISO	Past standard (1986)	Surface polishing	Surface area = 0.5 to 0.001 m^2	Deionized water Synthetic seawater Site water	Cylinder: unreactive, radiation resistant	10 to 20 cm	1, 3, 7, 10, 14, 28, 35, 42, 72, 102 days
ANSI/ANS 16.1(29) ANS (American Nuclear Society)	Standard (1986)	Surface washing	Cylinder, length/diameter = 0.2 to 5.0	Distilled water	Cylinder: unreactive material, sized to immerse sample	10 cm	2, 7 hours; 1, 2, 3, 4, 5, 14, 28, 43, 90 days
DLT(13)	Published research (1988)	Curing and surface washing	Cylinder, 4.5 cm diameter x 7.5 cm long	Distilled water	Large, wide mouth jar	Allows contaminant detection	0,1,4,7,24,31,48,72, 79,100 hours 0,4,24,31,72,104,168, 196 hours (latter for more immobile species)

mL/min. As with several of the other MCC tests, this test calls for the use of a reference test matrix to estimate the bias and within-laboratory precision of the method.

IAEA Dynamic Leach Test. In 1971, the International Atomic Energy Agency (IAEA) published a suggested standard for nuclear waste forms. This was in response to the wide variety of dynamic leach tests that were being performed in individual laboratories, each designed to meet the specific and unique objectives of the researcher. The protocol met with consent in principle, but the standard was never put into practice. Instead, many versions of a modified IAEA test proliferated, and the radioactive waste community was no closer to establishing a standard test. Some of the inherent experimental difficulties with the IAEA method were the experimental limitations presented by the one-dimensional leaching configuration, whereby only one circular face was contacted by leachant; a test duration that was far too long to demonstrate compliance; and a leachant replacement schedule that was too infrequent in the latter stages of the test.

ISO Leach Test. The International Standards Organization (ISO) followed more or less in the footsteps of IAEA in developing a standard leach test that was intended to serve as the basis for interlaboratory comparisons. This test did not simulate disposal-site conditions, such as very high pressures and temperatures. Therefore, it could not be used to make quantitative long-term extrapolations to determine the durability of products in repositories. This test has been largely replaced by ANSI/ANS 16.1.

ANSI/ANS 16.1. This voluntary standard of the American National Standards Institute (ANSI) was developed by the American Nuclear Society (ANS) to provide a common basis for evaluating the leachability of solidified nuclear waste forms. It was developed in response to the deficiencies of the original IAEA leach test. It is a simple, short-term test used to assess the quality of monolithic waste forms containing low level radioactive waste by measuring and indexing radionuclide release under controlled, reproducible conditions in a well-characterized leachant. This test was not intended to study the long-term leaching behaviour of these waste forms under actual disposal conditions. It is characterized by intermittent renewal of the leachant. A monolithic cylinder is suspended in demineralized water and frequent replacements of the leachant are made during the first week, with less frequent replacements for up to 90 days. Recently, the test has been widely used to study the leachability of nonradioactive contaminants.

DLT. The Dynamic Leach Test (DLT) is based on ANSI/ANS 16.1, and was modified to test solidified hazardous waste. The modifications include a revised leachant renewal

frequency, selected to maintain a driving force for leaching conditions in the leachate while allowing for analytical detection of the contaminants in the leachate. DLT provides two leachant renewal schedules, for mobile and immobile species.

5.2.3 Flow-through Tests (Table 13). Column and lysimeter studies tend to be selected for research purposes since they lend themselves to a better approximation of field conditions. As well, they are not readily applied in a regulatory framework because of their lengthy duration and often poor reproducibility. Therefore, no great efforts have been made to standardize these tests, particularly in the case of lysimeters. There are hundreds of column and lysimeter studies reported in the literature, which have been used to evaluate contaminant attenuation in soils and waste leaching. Two standard column protocols are discussed below, followed by several examples of lysimeter studies.

TABLE 13 FLOW-THROUGH TEST PROTOCOLS

Test name:	Column extraction method[30]	SLT, Column Method[25]
Proponent:	ASTM	SOSUV, Netherlands
Status of development:	Proposed standard	Published research
Leaching vessel:	4 in. (10 cm) diameter x 12 in. (30 cm) long cast acrylic column	5 cm I.D., 20 cm long column
Sample preparation:	Moisture, density, and particle size; adjustment to represent field conditions; curing and presaturation	Drying
Sample mass:	5000 g	-
Leachant:	ASTM Type IV water	Deionized water, pH = 4 with HNO_3
Liquid-to-solid ratio:	To simulate field conditions	10:1 at final collection
Percolation:	Forced upflow	Forced upflow
Leachate collection:	1st, 2nd, 4th, and 8th pore volume	Seven fractions/20 days
Leachate separation:	Optional filtration	-

ASTM Column Method. This draft method is a proposed laboratory procedure for generating aqueous leachates from inorganic, nonmonolithic waste materials in a column apparatus. It is not intended to produce results from which an engineered landfill could be designed, nor to provide the basis for classifying wastes based on their leaching characteristics. However, the waste samples should be physically, chemically, and biologically representative of the in situ waste. The column is prepared in a manner that simulates field conditions as closely as possible, and it is operated in a continuous upflow mode. As many pore volumes as required to establish meaningful trends or steady-state conditions are collected.

SOSUV SLT, Column Test. This column test forms part of a comprehensive set of leachability evaluation procedures, developed primarily for coal and municipal solid waste combustion residues, for use in the Netherlands (see SLT, Cascade Test). The test is performed when complete residue characterization is required or if a percolation system is to be simulated. Leachate is collected on a schedule to represent specified cumulative liquid-to-solid ratios. This test can be of short or long duration depending on research needs.

Lysimeter studies. Leach tests conducted in lysimeters are suited to site-specific modelling and the establishment of empirical data bases on the leachability of common waste streams, such as fly ash, municipal solid wastes, and mine tailings. If properly designed, they have the advantage of eliminating or reducing laboratory-scale problems associated with container-wall effects, sample preservation, subsampling, and sample heterogeneity. However, it is often difficult to maintain instrumentation, and to monitor and verify that internal conditions are, in fact, representative of the field, over the long term.

Of the many lysimeter studies reported in the literature, a few have been selected here as being typical of the objectives, setup, monitoring, and results obtained from experiments that are normally run over several years.(31-39)

5.2.4 Soxhlet Tests (Table 14).

MCC-5. This is a short-term, extreme-condition test designed to rapidly compare the leaching behaviour of monolithic radioactive waste forms. Since the conditions of the test will never be encountered in the field, site-specific predictions cannot be made. This test measures the normalized elemental mass losses from monolithic samples under ambient pressure in a leachant that is continuously redistilled from boiling water.

TABLE 14 SOXHLET TEST PROTOCOL

Test name:	MCC-5S(40) Soxhlet Test Method
Proponent:	Materials Characterization Center
Status of development:	Standard method
Leaching vessel:	Teflon soxhlet apparatus that allows complete immersion at all times
Sample preparation:	Fabricated individually and surface cut and washed
Sample mass:	N/A
Leachant:	Distilled and repository waters
Liquid-to-solid ratio:	Surface area/volume (volume of leaching vessel) = 0.0100
Flow rate:	0.2 mL/min
Percolation:	Soxhlet
Leachate separation:	Filtration through 0.45 μm membrane filter

Therefore, the temperature depends on the ambient pressure. The test can be run for 3 or 14 days, and sampling is performed at the end. Standard reference material is not yet available for this procedure. MCC-5 differs from the conventional soxhlet test in that the specimen is completely submerged in liquid at all times, using an overflow leaching container. Also, test conditions are better controlled, thus producing more reliable results than with the conventional soxhlet test.

6 ACCELERATION PROCEDURES

Under ideal conditions, long-term predictions of waste leaching would be made by mathematical models calibrated with short-term laboratory tests. Unfortunately, it is impossible to derive mechanistic relationships for all of the complex interactions of chemical and physical phenomena that constitute leaching. This limitation has led to the use of acceleration procedures in leaching tests.

An acceleration mechanism speeds up the leaching process, with respect to field conditions, without, in theory, altering the mechanisms of leaching. The time scale upon which measurements are taken, therefore, is compressed.

Both extraction and dynamic tests can be accelerated by controlling variables such as the surface area of contact, liquid-to-solid ratio, agitation, flow rate, leachant composition, and temperature.

In extraction tests, acceleration procedures, such as crushing and agitation, are used to reach steady-state conditions more quickly by eliminating mass-transfer limitations. Other variables can also be changed (higher liquid-to-solid ratio, and the use of a strong chemical solution) to study the effect of a change in the chemical environment on leaching.

In dynamic tests, acceleration procedures are aimed at observing rates of leaching, as opposed to the final steady-state condition. Variables that can be used to accelerate dynamic tests include temperature, flow rate, and leachant composition.

There are no hard and fast rules to design and perform accelerated tests. A test that incorporates an acceleration mechanism is run under the assumption that the mechanisms of leaching remain unaltered. This requires verification, however, since certain mineralogical alterations and changes in pore-water chemistry speed up leaching, but may not occur under natural conditions.

Concerns about the validity and utility of accelerated tests are twofold: 1) laboratory modification may change the actual mechanisms of leaching that are operational in the field, and 2) for a given rate-limiting mechanism, there may be no possible modification of the test conditions capable of causing acceleration.

The following limitations should be considered before selecting an accelerated leaching test:

- increased flow rates may cause erosion of the matrix as opposed to chemical dissolution

- increased temperatures may induce phase changes (e.g., dehydration of cement) and other chemical reactions
- many chemical, biological, and photolytic transformations cannot be accelerated by any method

7 INTERPRETATION CONSIDERATIONS

The results of leaching tests are obtained as laboratory leachate concentrations that can sometimes be used directly or need to be transformed to meet the test objectives listed in Table 1.

Since direct laboratory leachate concentrations are a function of the testing conditions, they cannot normally be interpreted as field leachate concentrations. However, concentrations obtained from extraction tests are suitable as relative criteria for the following objectives listed in Table 1: identification of leachable constituents, classification of hazardous wastes, evaluation of process modifications, and comparison of waste treatment methods.

Estimated field leachate concentrations can best be obtained from dynamic tests (e.g., column or lysimeter) when the hydraulic regime mimics field conditions. These data are difficult and costly to collect, but, fortunately, they are only required for two of the objectives listed in Table 1: design of leachate treatment systems and field concentration estimates.

Leaching test concentrations are often used to calculate other parameters, such as release, leach rate, and flux (defined below), to allow comparisons to be made among testing conditions (e.g., different liquid-to-solid ratios) to be used for engineering design (e.g., leachable mass of a contaminant per mass of waste), or to serve as input in mathematical models. These data conversions are useful for all of the objectives listed in Table 1.

This chapter addresses four elements of interpretation: data examination, data reduction, time-independent analysis, and time-dependent analysis.

7.1 Leachate Data Examination

Quality assurance requirements should be considered when a leaching study program is defined. Before the leachate data can be interpreted with respect to the objectives of the testing program, a number of routine examination steps should be applied to the results of the leaching tests:

1) Determine the precision and accuracy of the results by comparing replicates, examining the results of blanks and spikes, and comparing measured levels to analytical detection limits (analytical precision normally decreases as the measured values approach the detection limit).
2) Compare the results with published data for similar wastes if available.

3) Examine the gross ionic chemistry of the leachate. Constituents that are not necessarily contaminants, such as chloride, may provide insight into the nature of the solid waste and the leaching mechanism.

4) Try to determine whether simple factors have controlled the observed concentrations or release rates, and, if so, which ones. Consider leachate pH, redox potential, and ionic strength (as indicated by conductivity). Concentrations can be used to verify solubility products of simple compounds (e.g., hydroxides, sulphides). In more complex situations, chemical equilibrium models of the leachate can be developed. True chemical equilibrium can be verified by preparing a leachate oversaturated with respect to the leached species, continuing the test, and observing whether the same equilibrium state is reached.

5) If a relatively complete characterization of the leachate is available, compare the sum of the masses of individual constituents measured with the total dissolved solids, and verify electroneutrality by a charge balance on the equivalents of cations and anions. This may reveal that major constituents were not analyzed in the leachate.

Along with field verification and historic site evaluation, leached specimen examination is used to confirm leaching test results or to generate kinetic information through surface examination or leached layer profile measurement.

Direct examination of the leached specimen has mainly been applied to engineered waste forms (i.e., glass and cement-based solidified wastes) to establish the nature and extent of depletion of contaminants and to verify leaching test results.

Some micromorphological tools that may be used to examine leached specimens are listed in Table 15.

7.2 Leachate Data Reduction

The results of leaching tests are obtained as concentrations in the leachate. Reduction and transformation of these data are necessary to interpret the results. Various methods of expressing the results are listed below.

For both extraction and dynamic tests, the results can be expressed in the following manner:

- concentration: mass of constituent in leachate per mass or volume of leachate

TABLE 15 MICROMORPHOLOGICAL CHARACTERIZATION

X-ray diffraction	Used to identify mineralogical changes in the leached layer
Electron microscopy	Used to characterize the microstructural, morphological, and chemical changes induced as a result of leaching
Autoradiography	In the case of radioactive contaminants, autoradiographic analysis of the leached specimen will provide qualitative information on the extent of radionuclide depletion in the leached zone, relative to the unleached part of the specimen It may also provide important information on the distribution and partitioning of radionuclides in the cement matrix as well as between the cement matrix and aggregates

- release: mass of constituent in leachate per total mass of raw waste sample (must specify wet- or dry-weight basis)
- fraction leached: mass of constituent in leachate per mass of constituent in waste sample

For dynamic tests only, the results can be expressed in the following manner:

- leach rate: mass of constituent per unit time
- flux: mass of constituent per unit surface area and unit time
- cumulative release: cumulative mass of constituent in leachate per total mass of waste sample (must specify wet- or dry-weight basis)
- cumulative fraction leached: cumulative mass of constituent in leachate per mass of constituent in waste sample

It should be noted that expressing results as the fraction leached normally requires that the original concentration of the constituent in the specimen be known. Similarly, to express the results as a flux, the surface area of contact between the leachant and the specimen must be estimated.

Calculation of the release based on the raw waste mass is an appropriate method of expression for comparing leachability before and after treatment with stabilization/solidification additives, which act to lower the concentration of contaminants originally present in the raw waste sample.

In addition to the methods of expression listed above, leaching test results are often interpreted using mechanistic models to back-calculate parameters such as diffusivity, tortuosity, partition coefficients, and thermodynamic and kinetic parameters.

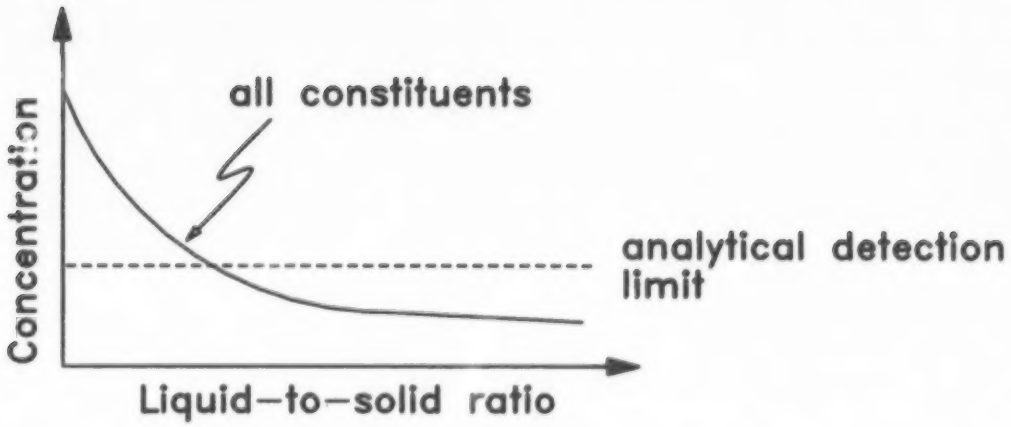
7.3 Time-independent Analysis

Given the fact that laboratory leaching tests do not normally simulate field conditions, it is desirable to attempt to obtain results that would be representative of worst-case conditions. In that respect, two performance parameters of interest are maximum concentration and maximum release.

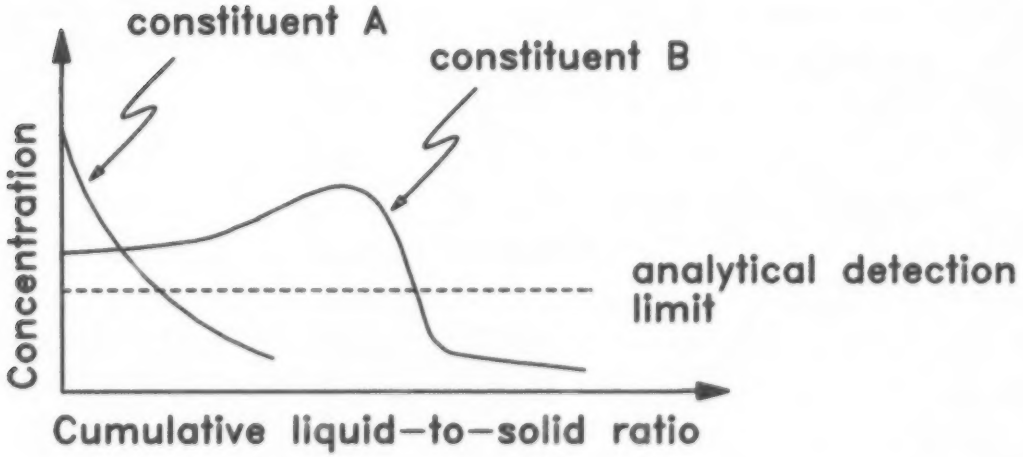
Maximum concentration and maximum release values may be obtained from either extraction tests or dynamic tests, but normally not under the same test conditions. Both performance parameters should be obtained from tests in which mass-transfer limitations are reduced or eliminated (i.e., particle-size reduction). The chemistry of the waste-leachant system and volume of the leachant are more important than kinetic considerations in determining the maximum possible concentration and release values.

Figure 14 shows typical curves for leachate concentrations as a function of the liquid-to-solid ratio for extraction and dynamic tests. For an extraction test (Figure 14a), the concentration is greatest at low liquid-to-solid ratios (i.e., concentration in the pore fluid) and steadily decreases as the liquid-to-solid ratio is increased. For very high liquid-to-solid ratios, the concentration in the leachate may drop below the analytical detection limit, a condition that should be avoided. Low leachate concentrations at low liquid-to-solid ratios are desirable in terms of environmental protection, and indicate that contaminants are well contained.

For a dynamic test (Figure 14b), the concentration generally decreases as the cumulative liquid-to-solid ratio increases (constituent A). Since the leached constituents are removed from the leaching vessel, the concentration normally drops below the analytical detection limit at a lower cumulative liquid-to-solid ratio than in an extraction test. Preferential leaching of one constituent prior to another is often observed in dynamic tests. This is illustrated in Figure 14b when constituent B is released after constituent A has been leached from the waste (e.g., an increase in metal solubility after



a) Extraction test



b) Dynamic test

FIGURE 14 TYPICAL CONCENTRATION CURVES

the buffering capacity has been removed). In that situation, a maximum concentration may be observed at a relatively high cumulative liquid-to-solid ratio.

Figure 15 shows typical curves for the release of constituents as a function of the liquid-to-solid ratio for extraction and dynamic tests. Release is positively correlated to the liquid-to-solid ratio, and increases to an asymptotic maximum. It may be impractical to obtain maximum release from an extraction test because dilution may result in concentrations below the analytical detection limit (e.g., Figure 14). This problem may be alleviated in dynamic tests (Figure 15b), since data are obtained as the cumulative liquid-to-solid ratio increases. Preferential leaching is also exemplified in Figure 15b by the sudden increase in the cumulative release of constituent B after most of constituent A has been leached.

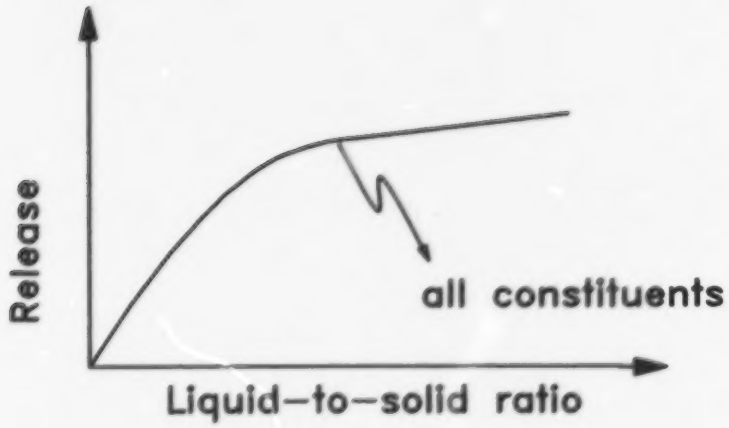
Both concentration and release are strongly affected by the nature of the leachant. Generally, for mild leachants are low liquid-to-solid ratios, the leachate chemistry is controlled by the waste. For aggressive leachants and/or higher liquid-to-solid ratios, leachate chemistry is controlled by the leachant and the test becomes closer to a chemical extraction.

Aggressive leachants are commonly used to neutralize the waste and inhibit preferential leaching. A common practice, for example, is to use an acidic leachant to neutralize excess alkalinity and force the leaching of pH-sensitive constituents. Care should be taken in selecting aggressive leachants to relate the nature of the leachant to liquids that may possibly contact the waste under field conditions. A series of chemical solutions may be used as leachants to remove from 0 to 100% of a constituent from a waste (Figure 16).

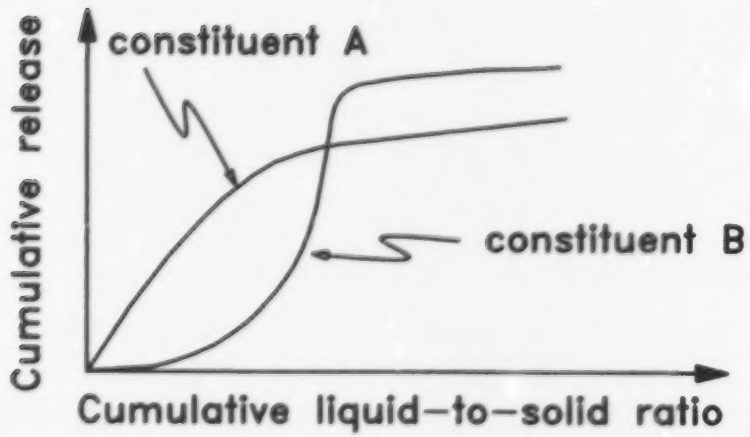
7.4 Time-dependent Analysis

When time is a variable of interest, mass-transfer limitations become important. For this reason, tests designed to provide temporal information must not affect a waste form's physical integrity. These considerations are not limited to waste forms that are monolithic and have high physical strength. Granular or soil-like wastes may be emplaced and compacted in the field to obtain physical and engineering properties (density, hydraulic conductivity, etc.) that may determine the hydraulic regime of the leachant.

Temporal information can often be derived from flow-around and flow-through tests. Flow-around tests are more easily interpreted, however, due to the absence of advection through the waste. As a result, flow-around tests have become the instrument



a) Extraction test



b) Dynamic test

FIGURE 15 TYPICAL RELEASE CURVES

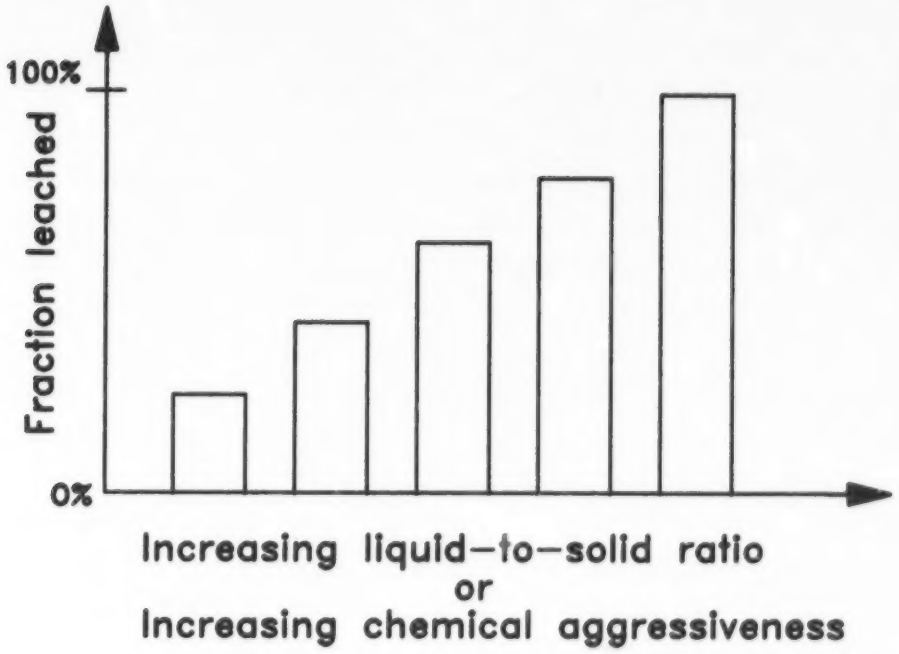


FIGURE 16 FRACTION LEACHED ACHIEVABLE WITH DIFFERENT LEACHING TESTS

of choice (especially in the nuclear waste industry) to study engineered waste forms. A large number of mathematical models are available to assist in the interpretation and extrapolation of the results of flow-around tests. Flow-through tests are still important for studying more permeable wastes, such as ashes, slurries, and contaminated soils. Although the simple conditions of flow-around tests allow for the use of analytical models, more complex numerical solutions are necessary to interpret the results of flow-through tests. Some interpretation considerations for flow-around tests are presented below.

Flow-around test data are normally expressed as a flux or a cumulative fraction leached (either of which can easily be converted from one to the other) as a function of time. The rate of leachant renewal controls the concentration in the leaching vessel (a boundary condition) and determines both the driving force for leaching and the type of mathematical model that may be used to interpret the results. The rate of leachant renewal may be expressed as a leachant velocity, v, equal to the volume of leachant contacting the waste per unit surface area of waste per unit time, i.e.,

$$v = (\text{volume}/\text{area})/\text{time} = \text{distance}/\text{time}$$

The effect of the hydraulic regime of the leachant on the flux can be visualized by considering the dependency of the flux on the leachant velocity (Figure 17).

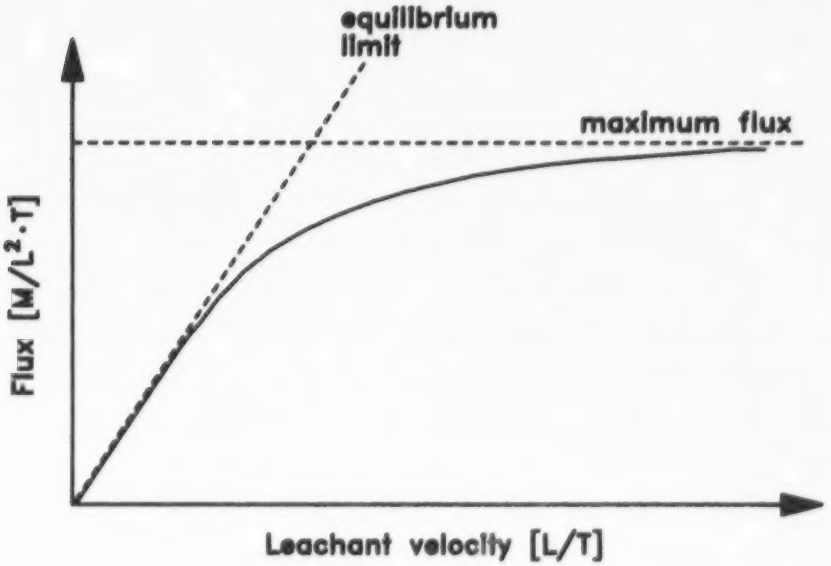


FIGURE 17 EFFECT OF LEACHANT VELOCITY ON THE LEACHING RATE

The slope of the curve in Figure 17 has the dimensions of a concentration. The curve has two asymptotic limits at which the slope can be interpreted as the concentration of the leached species near the interface. The maximum flux is reached when the leachant velocity tends towards infinity. Under such flow conditions, there is no accumulation of leached species in the leachate (the slope of the curve tends towards zero), and the leaching driving force is at a maximum. The other limit corresponds to the saturation concentration of a species under the given leaching conditions. This limit can be approached for tests conducted under a sufficiently low leachant velocity.

The two limits correspond to tests that are widely used. The ANSI/ANS 16.1 protocol and family of tests use a high leachant velocity and, for the purposes of interpretation, assume that the boundary condition (i.e., the leachate concentration at the waste-leachant interface) is zero. This condition leads to the measurement of the highest possible flux, but poses some problems, from a practical point of view, in precisely measuring the amount leached since the concentration in the leachate is often close to the analytical detection limit. Kinetic information may also be derived from the MCC-1

protocol by monitoring the approach to saturation. Flow-around tests using a leachant velocity that falls between these two limits are more difficult to interpret and require the use of numerical methods.

A simple empirical model commonly used to interpret the results of flow-around tests, including several rate-limiting leaching mechanisms, is

$$CFL(t) = a + bt^{1/2} + ct$$

where CFL(t) is the cumulative fraction leached, t is time, and a, b, and c are constants. The first term, a, is associated with initial washing phenomena, or the rapid dissolution of soluble species present on the surface. The second term, $bt^{1/2}$, describes diffusion-limited transport processes. When CFL is plotted against the square root of time, a straight line is an indication that diffusion is limiting the flux. A number of leaching mechanisms may be associated with the third term, e.g.,

- slow, kinetically controlled mobilizing reactions (such as redox reactions)
- corrosion or matrix dissolution
- dissolution as a function of a species present in the leachant

The empirical equation does not lend itself to extrapolation over time because it does not allow for positive identification of the rate-controlling mechanisms. It can be used, however, as a screening tool prior to the development of more sophisticated models.

8 CONCLUSIONS

Several objectives were identified for conducting leaching tests, ranging in complexity from the simple identification of leachable species to risk assessment with respect to land disposal of wastes. To meet these objectives, a literature survey revealed the existence of a large number of protocols that were classified based on experimental characteristics. Twenty-six protocols, either standards or accepted research tools, were described in detail. It is clear that meeting a testing program's objective(s) may require the use of different tests to obtain complementary information.

The ultimate goal of performing leaching studies is to evaluate the impact of land disposal of solid wastes. Since leaching phenomena are slow, designers of short-term laboratory studies are faced with the challenge of generating information at an accelerated rate or producing information that can be used in mathematical models for extrapolation. Both approaches have severe limitations, as summarized below.

Laboratory leaching procedures that attempt to simulate field conditions are not appropriate to predict long-term leachability. Even if a leaching test existed that would perfectly simulate field conditions, it would only represent a time period equivalent to the test duration. On the other hand, acceleration procedures (by controlling variables such as the surface area of contact, liquid-to-solid ratio, agitation, flow rate, leachant composition, and temperature) may cause matrix alteration (such as phase changes), introduce unwanted mechanisms (such as erosion), and eliminate mitigating effects (such as a redox environment controlled by biological activity), producing results that would never be observed under field conditions.

Extrapolation through mathematical models also has severe limitations. Most models only consider one chemical species and are based on the assumption that the waste's morphological properties are constant over time, conditions that are seldom met in practice. Furthermore, there is growing evidence that interfacial reactions between the waste and the surrounding material (normally two markedly different chemical environments) may alter the transport properties of the media and drastically reduce leaching in the long term.

This compendium may leave the reader unsatisfied after raising more questions than it answers. Its main purpose, however, is to present a brief introduction to leaching tests as a source of reference to assist in the selection of appropriate methods for particular applications. An example of its usefulness can be found in Proposed Protocol for Stabilized/Solidified Wastes,[42] in which the rationale for the selection and

interpretation of leaching tests to manage solidified wastes is given for four different utilization and disposal scenarios.

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